Sampling and Analysis Plan for
Petro-Chemical Systems, Inc. (Turtle Bayou) Site
Liberty County, Texas
EPA Identification No. TXD980873350

Remedial Action Contract 2 Full Service
Contract: EP-W-06-004
Task Order: 0134-RARA-0681

Prepared for

U.S. Environmental Protection Agency
Region 6
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20 March 2019

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LIST OF ACRONYMS AND ABBREVIATIONS

ARCO	Atlantic Richfield Company
bgs	Below ground surface
CLP	Contract Laboratory Program
CR	County Road
CRDL	Contract-required Detection Limit
CRQL	Contract-required Quantitation Limit
DQA	Data quality assessment
DQO	Data quality objective
DPT	Direct-push technology
EA	EA Engineering, Science, and Technology, Inc., PBC
EDD	Electronic data deliverable
EPA	U.S. Environmental Protection Agency
EPEC	EPEC Polymers Inc.
ESD	Explanation of Significant Differences
GC	Gas chromatograph
GPS	Global Positioning System
HASP	Health and Safety Plan
IDW	Investigation-derived waste
ISCO	*In situ* chemical oxidation
LCS	Laboratory control sample
Lyondell	Lyondell Chemical Company
MCL	Maximum Contaminant Level
MD	Matrix duplicate
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OU	Operational unit

LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)

PARCCS	Precision, accuracy, representativeness, completeness, comparability, and sensitivity
PPE	Personal protective equipment
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
RA	Remedial action
RAC	Remedial Action Contract
RD	Remedial design
ROD	Record of Decision
RPD	Relative percent difference
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SIM	Selective ion monitoring
Site	Petro-Chemical Systems, Inc. (Turtle Bayou) Site
SOP	Standard operating procedure
SOW	Statement of Work
SVOC	Semivolatile organic compound
TCEQ	Texas Commission on Environmental Quality
TCLP	Toxicity characteristic leaching procedure
UAO	Unilateral Administrative Order
USACE	U.S. Army Corps of Engineers
USCS	Unified Soil Classification System
VOC	Volatile organic compound

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1. PROJECT DESCRIPTION AND MANAGEMENT

EA Engineering, Science, and Technology, Inc., PBC (EA) has been authorized by the U.S. Environmental Protection Agency (EPA), under Remedial Action Contract (RAC) No. EP-W-06-004, Task Order 0134-RARA-0681, to perform a Remedial Action (RA) at the Petro-Chemical Systems, Inc. (Turtle Bayou) Site (Site) near Liberty, Liberty County, Texas. EA has prepared this Sampling and Analysis Plan (SAP) in accordance with: (1) specifications provided in the EPA Statement of Work (SOW), received on 18 November 2015 (EPA 2015) and (2) the approved Remedial Work Plan for Petro-Chemical Systems, Inc. (Turtle Bayou) Superfund Site (Work Plan), Revision 01 (EA 2018), submitted on 5 July 2018.

This SAP is a combination Quality Assurance Project Plan (QAPP) and Field Sampling Plan that details sample collection procedures and analytical methods required to collect sufficient data to perform RA activities at the Site. Combining these two standard deliverables into a single document allows the EA team to streamline the planning process, while ensuring the data collected is of sufficient quality for its intended use.

This SAP was prepared in conjunction with the Health and Safety Plan (HASP), Revision 01 (EA 2016), which together, present the overall approach for implementing the technical assistance field program. The HASP specifies employee training, protective equipment, personal air monitoring procedures, medical surveillance requirements, standard operating procedures, and contingency planning procedures.

This SAP was prepared in accordance with EA's Quality Management Plan (EA 2014) and meets requirements set forth in *EPA Requirements for Quality Assurance Project Plans* (EPA 2001) and *EPA Guidance for Quality Assurance Project Plans, QA/G-5* (EPA 2002).

This SAP describes procedures to assure that the project-specific data quality objectives (DQOs) are met, and that the quality of data (represented by precision, accuracy, representativeness, completeness, comparability, and sensitivity [PARCCS]) is known and documented. The SAP presents the project description, project organization and responsibilities, and quality assurance (QA) objectives associated with the sampling and analytical services to be provided in support of RA at the Site. Table 1 demonstrates how this SAP complies with the elements of a QAPP currently required by EPA guidance (EPA 2001, 2002).

The overall QA objectives are as follows:

- Attain quality control (QC) requirements for analyses specified in this SAP.
- Obtain data of known quality to support goals set forth for this project.
- Document all aspects of the quality program including performance of the work and any required changes to work at the Site.

The EPA Region 6 Contracting Officer is Mr. Brian Delaney. The EPA Region 6 Project Officer is Mr. William G. Johnson, Jr. The EPA Region 6 Task Order Monitor, Ms. Raji Josiam, is

responsible for the project oversight. EA will perform all tasks under this Task Order in accordance with this SAP. The EA Project Manager, Ms. April Ballweg, is responsible for implementing all activities required by this Task Order. Figure 1 presents the proposed project organization for this Task Order.

1.1 SITE BACKGROUND AND PROBLEM DEFINITION

This section describes the following:

- Site background and description (Section 1.1.1)
- Problem definition (Section 1.1.2).

1.1.1 Site Background and Description

The Site is located on County Road (CR) 126 (also known as Frontier Park Road), south of Liberty, Liberty County, Texas (Figure 2). The Site appears to have been used to dispose of unpermitted waste starting in the late 1960s. The disposal of waste at the Site was documented in the Texas Water Quality Board records as early as 1971. Records indicate the dumping of waste oils in unlined pits and on Frontier Park Road. The Site was not authorized as a waste disposal facility; therefore, the exact nature of the substances disposed of at the Site is uncertain. According to EPA, it appears that the waste was dumped indiscriminately from trucks at numerous locations, and that the waste disposal activities continued after 1971.

In 1971, an application for a commercial industrial waste disposal permit with the name Petro-Chemical Systems, Inc. was filed with the State of Texas. The application included a Site Development Plan. In response to the application, local citizens organized to oppose the application. After public hearings were held and additional information was evaluated in response to a citizen's lawsuit, the State's approval for the application was withheld. In 1974, Petro-Chemical System, Inc., withdrew the application.

After 1974, the Site was subdivided into 5-acre and 15-acre plots and sold for residential development. Residential use of the Site has been continuous since 1974, except during previous remedial activity on Frontier Park Road completed in August 1988, during which Site residences were temporarily relocated. No residents live on any of the identified disposal areas; however, six families live adjacent to waste disposal areas: the CR 126 West Area, the Easement Area, and the Bayou Disposal Area.

EPA proposed the Site for inclusion on the National Priorities List (NPL) in 1984, and the Site was placed on the NPL in 1986.

The Site was previously divided into two operable units (OUs). OU 1 included contaminated soil along CR 126 (Frontier Park Road). OU 2 included several areas of contaminated soil and groundwater, including the West Road Area, Office Trailer Area, Main Waste Area, Easement Area, and Bayou Disposal Area. Subsequent investigations identified additional areas of contaminated soil and groundwater: the CR 126 West Area and the MW-109 Area.

EPA signed the OU 1 Record of Decision (ROD) on 27 March 1987, which prescribed excavation of contaminated soil along CR 126 (Frontier Park Road); placement of the contaminated material in a temporary Resource Conservation and Recovery Act storage facility located in the Main Waste Area; and backfill and paving of the roadway. EPA implemented the RA, which was completed in August 1988.

EPA signed the OU 2 ROD on 6 September 1991, which prescribed a variety of remedial alternatives to control source areas and contaminated groundwater within OU 2. On 22 December 1993, EPA issued a Unilateral Administrative Order (UAO) to Lyondell Chemical Company (Lyondell) and Atlantic Richfield Company (ARCO) to implement the remedial design (RD) and RA for OU 2. The remedial operations initially began as field pilot studies to support RD activities and expanded into full-scale remediation systems. The primary technologies used at the Site were groundwater extraction with nutrient-and-oxygen amended injection (*in situ* bioremediation) and soil vapor extraction. Other technologies applied in more recalcitrant areas included *in situ* thermal desorption (i.e., soil heating), *in situ* chemical oxidation (ISCO) using potassium permanganate injection, and bio-augmentation.

EPA signed the OU 2 ROD Amendment on 30 April 1998, which modified the remedial components for contaminated soil, incorporated additional remedy components for hot spot areas, modified the groundwater remedy, and incorporated monitored natural attenuation and institutional controls.

On 8 December 1998, EPA entered into a Consent Decree with Lyondell and ARCO, which superseded the provisions of the 1993 UAO, and transferred responsibility for remediation of the Bayou Disposal Area to EPA and the Texas Commission on Environmental Quality (TCEQ).

On 22 September 2006, EPA signed a second ROD Amendment for OU 2, which documented the technical impracticability of the prescribed contaminated soil and groundwater remedies and cleanup criteria. In general, the areal extent of the groundwater contamination was greatly reduced and a significant amount of contamination was removed. However, the ability of the remedial technologies to continue removing the remaining contamination declined significantly over time and eventually plateaued. While the remedial technologies have reduced the mass of contaminants, considerable mass remains at moderate concentrations. The contaminants, left primarily in the Site's clay and silty soils, continue to act as a source of contamination to the underlying groundwater. EPA had determined that in areas where significant disposal has taken place, complete restoration of groundwater is technically impracticable, and that the applicable or relevant and appropriate requirements for groundwater restoration should be waived.

The 2006 ROD Amendment also incorporated the CR 126 West Area and discussed the evaluation of the MW-109 Area for future consideration in a subsequent EPA decision document. On 21 August 2007, EPA entered into a Consent Decree with EPEC Polymers Inc. (EPEC), directing them to address the CR 126 West Area and Bayou Disposal Area. EPEC successfully implemented the remedy for the CR 126 West Area. For the Bayou Disposal Area, the remedy included leaving impacted soils in place, implementing institutional controls, and restricting land use to non-residential.

Based on the large volume of waste, it was not cost-effective to excavate and remove all impacted soil. It has also been demonstrated that groundwater remediation is not technically practicable since, once remediation is terminated, the contaminant concentrations in groundwater will rebound as long as a substantial portion of residual contamination remains in the soil.

Since the 1998 and 2006 ROD Amendments, a significant change in the Site's current and anticipated land use occurred for large portions of the Site. Specifically, for the Site's West Road Area, Main Waste Area, Office Trailer Area, and Easement Area, residential land use was no longer reasonably anticipated. Lyondell acquired these properties and currently restricts access to these areas, such that residential use on this property cannot occur. In regards to the CR 126 West Area and the Bayou Disposal Area, EPEC initiated contacts with landowners regarding sale or deed restrictions that allow EPEC to purchase the properties or groundwater rights and/or provide land use restrictions. Potential future exposures would likely be limited to road utility workers, trespassers, Site maintenance workers, and contractors involved in the groundwater monitoring program.

The MW-109 Area is an area of concern at the Site (Figure 2). The Explanation of Significant Difference (ESD) for the Site, dated 23 September 2010 (EPA 2010), documents the decision to perform a RA for contaminated soil at the MW-109 Area using ISCO and excavation with bio-treatment, as well as installation of three new monitoring wells.

MW-109 was a shallow groundwater monitoring well (30 feet below ground surface [bgs]) located approximately 1,000 feet east of the CR 126 West Area and approximately 300 feet west of the West Road Area along CR 126 (Figure 3). From August 2000 through May 2005, groundwater samples collected from MW-109 indicated elevated contaminant concentrations of benzene varying from non-detect to 13,000 parts per billion; however, the sample results indicated a decreasing trend.

In September 2000, Lyondell replaced a poorly constructed residential well with a new well. The new residential well has a completed depth of 186 feet bgs and was constructed to prevent the well from acting as a potential migration pathway for impacted groundwater from the shallow water-bearing zone into deeper water-bearing zones.

Analysis of groundwater samples collected from two nearby monitoring wells (MW-33 and MW-110) have not detected any contaminants of concern since August 2003. Analysis of a May 2005 sample collected from MW-108 showed a single detection of tert-butyl alcohol above the TCEQ's Protective Concentration Level. In April 2005, three temporary wells were installed near MW-109 and sampled. Analysis of the groundwater samples did not detect any volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), or Target Analyte List metals concentrations above respective Maximum Contaminant Levels (MCLs) (EPA 2009) or EPA Risk-based Regional Screening Levels (RSLs). Groundwater monitoring in the MW-109 Area indicates that the extent of groundwater contamination is limited. The terrain is largely wooded with low relief, and shallow groundwater is typically encountered between 12 and 15 feet bgs.

Additional studies were conducted in 2007 to evaluate the extent of soil contamination in the MW-109 Area. The evaluations found contaminated soil exceeding the soil cleanup criteria for benzene and naphthalene at depth intervals between 3.5 and 14.5 feet bgs. The October 2007 evaluation document indicated an estimated volume of 2,388 cubic yards of impacted soil in the MW-109 Area.

The 1998 ROD Amendment identified excavation with bio-treatment as a remedy for hot spots. Hot spots were areas where there was increased soil permeability above that found in background areas. Though the MW-109 Area did not meet the soil permeability criteria of 1×10^{-6} centimeters per second established for a hot spot, like several other areas of the site, it did meet the general requirements of a hot spot based on elevated benzene concentrations. Approximately 2,209 cubic yards of contaminated soil from the MW-109 Area were excavated and bio-treated onsite. This included impacted soils from 3 to 14 feet bgs. Soils within the 0 to 3-foot depth interval were demonstrated to meet the soil cleanup criteria (as identified in the 2006 ROD Amendment). Following bio-treatment, the soil that met the residential soil cleanup criteria was used to backfill the excavated areas, and the soil that met the non-residential (industrial) soil cleanup criteria was used to backfill the right-of-way area adjacent to the excavation.

In 2009, EPA entered into an interagency agreement with the U.S. Army Corps of Engineers (USACE) to implement the RD and RA at the MW-109 Area. This area was remediated using ISCO and excavation with bio treatment. ISCO was the remedy that was used for the nearby CR-126 West Area and was selected as the remedy for that area in the 2006 ROD Amendment.

Cleanup criteria from the previous ROD Amendments were applied at the MW-109 Area; in particular, residential soil cleanup criteria for the soils in the Residential Remediation Area and industrial soil cleanup criteria for the soils in the roadway right-of-way. Following several rounds of chemical injections in the MW-109 Area, soil samples were collected and evaluated. A reduction in benzene and naphthalene concentrations was observed; however, the results indicated that the three rounds of chemical injections originally indicated in the design would not be sufficient to achieve soil cleanup criteria in a timely and cost-effective manner, and that additional injection events were required to clean up the highly plastic clay soil. Hence, an alternate remedy of excavation and bio-treatment was implemented, as identified in the 1998 ROD Amendment.

Data collected within the MW-109 Area indicates that the extent of groundwater contamination is limited (based on the results of groundwater sampling conducted in surrounding permanent and temporary wells). As part of the 2010 ESD, USACE plugged and abandoned MW-109 and installed replacement well MW-109A, as well as the three new monitoring wells screened in the S1 sand: MW-191, MW-192, and MW-193 (Figure 3).

To help determine the nature and extent of contamination, quarterly groundwater sampling was completed by EA between April 2011 and January 2013 consisting of a total of eight groundwater monitoring and sampling events. Based upon a review of the groundwater analytical data, it is apparent that the majority of detected groundwater impact is limited to MW-109A, with principal contaminants of concern being benzene, naphthalene, and toluene. A

review of the groundwater analytical data, in conjunction with the predicted groundwater flow and spatial distribution of wells, indicates a potential data gap in the groundwater monitoring well network to the south of MW-109A. Due to the apparent lack of a downgradient groundwater monitoring well, a complete evaluation of the nature and extent of the contamination was not performed.

1.1.2 Problem Definition

The purpose of this Task Order is to provide EPA with technical support in evaluating the nature of the groundwater impact and delineating the extent of groundwater contamination in the MW-109A Area of the Site. EPA has tasked EA with the following:

- Installation of up to 20 temporary groundwater sampling points using direct-push technology (DPT)
- Collection and field screening of groundwater samples collected from the DPT sampling points
- Installation of up to six new monitoring wells
- Groundwater sampling of up to six new monitoring wells and up to seven existing monitoring wells
- Performance of one vapor intrusion assessment at a residence located within proximity of MW-109A Area.

EA will conduct a total of up to four separate mobilizations during the period-of-performance for this RA (which expires on 21 November 2020) to achieve the Task Order objectives (Section 2).

1.2 DESCRIPTION OF PROJECT OBJECTIVES AND TASKS

This section describes the project objectives and tasks for this SAP.

1.2.1 Project Objectives

The primary objective of this SAP is to collect sufficient groundwater and field screening data from monitoring wells and DPT sampling points to determine nature and extent of groundwater contamination in the MW-109A Area. In order to meet this objective, EA will install and sample up to six new monitoring wells and collect groundwater samples from up to seven existing monitoring wells in the MW-109A Area; up to two groundwater sampling events are anticipated to achieve objectives. Once the groundwater contamination is delineated, EA will use this information to support development of a Technical Impracticability Waiver Zone in the vicinity of the MW-109A Area.

EA will also conduct a vapor intrusion assessment at a private residence in close proximity to the MW-109A Area to determine if EPA RSLs for VOCs in residential indoor air quality are being met (EPA 2017).

1.2.2 Project Tasks

To complete the technical assistance activities, EA will perform the following tasks (with subtasks), which are outlined in the Task Order SOW (EPA 2015) and described in detail in Section 2:

- Project management
- Community involvement
- Management support
- Development and update of site-specific plans
- Sample management
- Groundwater sampling using DPT
- Monitoring well installation
- Monitoring well sampling
- Surveying of top-of-casing elevations for existing monitoring wells and recalculating potentiometric surface elevations and groundwater flow direction
- Inspection report
- RA Report.

EA's field activities will be conducted in accordance with this SAP to ensure proper sample management, including accurate chain-of-custody procedures for sample tracking, protective sample packaging techniques, and proper sample preservation techniques, as well as EA's site-specific HASP, Revision 01 (EA 2016). Sample management will be conducted using the EPA-approved "Scribe" software.

1.3 DATA AND MEASUREMENT QUALITY OBJECTIVES

The following subsections present the DQOs and measurement quality objectives identified for this project.

1.3.1 Data Quality Objectives

DQOs are qualitative and quantitative statements developed through the seven-step DQO process (EPA 2000a, 2006a). The DQOs clarify the study objective, define the most appropriate data to collect and the conditions under which to collect the data, and specify tolerable limits on decision errors that will be used as the basis for establishing the quantity and quality of data needed to

support decision making. The DQOs are used to develop a scientific and resource-effective design for data collection. The 7-step iterative process used to prepare the DQOs is as follows:

- Step 1 – State the Problem
  - — Define the problem that necessitates the study.
  - — Identify the planning team.
  - — Examine budget and schedule.
- Step 2 – Identify the Goal of the Study
  - — State how environmental data will be used in meeting objectives and solving the problem.
  - — Identify study questions.
  - — Define alternative outcomes.
- Step 3 – Identify Information Inputs
  - — Identify data and information needed to answer study questions.
- Step 4 – Define the Boundaries of the Study
  - — Specify the target population and characteristics of interest.
  - — Define spatial and temporal limits.
  - — Define the scale of inference.
- Step 5 – Develop the Analytical Approach
  - — Define the parameter of interest.
  - — Specify the type of inference.
  - — Develop the logic for drawing conclusions from findings.
- Step 6 – Specify the Performance or Acceptance Criteria
  - — Specify probability limits for false rejection and false acceptance decision errors.
  - — Develop performance criteria for new data being collected or acceptance criteria for existing data being considered for use.
- Step 7 – Develop the Plan for Obtaining Data
  - — Select the resource-effective sampling and analysis plan that meets the performance criteria.

The seven steps of the DQO process for this project are presented in Table 2.

1.3.2 Data Categories

In order to produce data suitable for decision-making, an appropriate analytical technique must be selected. The EPA Superfund program has developed two descriptive categories of analytical techniques: (1) field-based techniques and (2) fixed-laboratory techniques. The type of data

generated depends on the qualitative and quantitative DQOs developed for a project. Regardless of whether the data was analyzed utilizing field or laboratory techniques, it must be of adequate quality for the decision-making process for which it was collected.

For this project, field-based and fixed-laboratory data will be collected. Section 2 discusses the methods that will be used to analyze the samples. Only definitive analytical data will be used to support decisions made for this project. Field water quality parameters, including pH, specific conductance, dissolved oxygen, water temperature, and oxidation-reduction potential, and turbidity, will be measured and the field data recorded on groundwater data sampling sheets for DPT groundwater collection points and monitoring wells. Canister gauge pressure readings, leak test results, and environmental conditions observed at the time of vapor intrusion air sampling will be recorded on air sampling data sheets.

Rigorous analytical methods (e.g., EPA Contract Laboratory Program [CLP] methods) are used to generate analyte-specific, definitive data. The definitive quality of the data is assured by: (1) strict adherence to standard operating procedures (SOPs) and QC processes during data collection; (2) documented control and traceability of reference standards, calibrations, and instrument performance; and (3) acceptable performance of field and laboratory QC procedures within the defined limits established for these procedures.

EA anticipates using the EPA Region 6 Laboratory and/or CLP for analysis of groundwater samples collected during up to four rounds of sampling anticipated during the period-of-performance. EA intends to use a subcontractor analytical laboratory for analysis of those parameters that cannot be analyzed by the EPA Region 6 Laboratory and/or CLP, such as air and investigation-derived waste (IDW) samples. If EPA Region 6 Laboratory and/or CLP are unable to meet project schedule requirements, EA will use a subcontractor analytical laboratory for the water sample analyses as well.

1.3.3 Measurement Quality Objectives

Groundwater and air analytical results will be evaluated in accordance with PARCCS parameters to document the quality of the data and to ensure that the data are of sufficient quality to meet the project objectives. Of these PARCCS parameters, precision and accuracy will be evaluated quantitatively by collecting the QC samples listed in Table 3.

The subsections below describe each of the PARCCS parameters and how they will be assessed within this project.

1.3.3.1 Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions. Usually, combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically as a relative percent difference (RPD).

RPD is calculated as follows:

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100\%$$

where: A = first duplicate concentration
B = second duplicate concentration

Field sampling precision is evaluated by analyzing field duplicate samples. For every 10 aqueous or air samples collected, one field duplicate sample will be collected. Field instruments will be calibrated each day following manufacturer recommendations prior to daily use.

Laboratory analytical precision is evaluated by analyzing laboratory duplicates, matrix duplicates (MDs) or matrix spikes (MS) and matrix spike duplicates (MSDs). For this project, MS and MSD QC samples will be generated in association with aqueous sample analysis. The results of the analysis of the MS/MSD samples will be used to calculate the RPD as a measure of lab precision. MS/MSD samples will not be analyzed for air samples. Laboratory precision for air analysis will be determined using either laboratory control samples or laboratory duplicate samples.

1.3.3.2 Accuracy

A program of sample spiking will be conducted to evaluate laboratory accuracy. This program includes analysis of the MS and MSD samples, laboratory control samples (LCSs) or blank spikes, surrogate standards, and method blanks. MS and MSD samples will be prepared and analyzed at a frequency of 5 percent for water samples. LCSs or blank spikes are also analyzed at a frequency of 5 percent for both air and water. Surrogate standards, where available, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

$$\text{Percent Recovery} = \frac{S - C}{T} \times 100\%$$

where: S = measured spike sample concentration
C = sample concentration
T = true or actual concentration of the spike

The objective for accuracy of field measurements is to achieve and maintain factory specifications for the field equipment. The water quality meter (for groundwater samples only) should be calibrated with certified calibration standard solutions at the start of each field day.

1.3.3.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an

environmental condition that they are intended to represent. For this project, representative data will be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will also be ensured through the consistent application of established field and laboratory procedures. Field blanks (if appropriate) and laboratory blank samples will be evaluated for the presence of contaminants to aid in evaluating the representativeness of sample results. Data determined to be non-representative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

1.3.3.4 Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability are exceeded. When all data validation is completed, the percent completeness value will be calculated by dividing the number of useable sample results by the total number of sample results planned for this investigation.

As discussed further in Section 2.11, completeness will also be evaluated as part of the data quality assessment process (EPA 2000b). This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected.

1.3.3.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data. Standard EPA analytical methods and quality control will be used to support the comparability of analytical results with those obtained in other testing. Calibrations will be performed in accordance with EPA CLP or other industry standard method-specific criteria or manufacturer's specifications.

1.3.3.6 Detection and Quantitation Limits (Sensitivity)

The analytical parameters and their quantitation limits for use on this project are determined under the EPA CLP SOW (EPA 2016). Groundwater samples collected from the monitoring wells will be analyzed in accordance with CLP methodology. The Contract-required Detection Limit (CRDL) is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method. The quantitation limit represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a sample matrix. Contract-required Quantitation Limits (CRQLs) are contractually specified maximum quantitation limits for specific analytical methods and sample matrices, such as soil or water, and are typically several times the method detection limit (MDL) to allow for matrix effects.

For this project, analytical methods have been selected so that the CRQL for each target analyte is below the applicable regulatory screening criteria, the MCLs and/or RSLs, where ever practical. Samples results for the CLP analyses will be reported as estimated values if

concentrations are less than the CRQL but greater than CRDL. The CRDL for each analyte will be listed as the detection limit in the laboratory's electronic data deliverable (EDD). The results for the air sample analysis will be reported by the laboratory as either 1) detections above the method reporting limit, 2) detections below the method reporting limit and above the detection limit (J-flagged data) and 3) results that are non-detect below the method reporting limit.

1.4 SPECIAL TRAINING AND CERTIFICATION

This section outlines the training and certification required to complete the activities described in this SAP. The following sections describe the requirements for the EA team and subcontractor personnel working onsite.

1.4.1 Health and Safety Training

EA field team personnel who work at hazardous waste project sites are required to meet the Occupational Safety and Health Administration (OSHA) training requirements defined in 29 Code of Federal Regulations 1910.120(e). These requirements include: (1) 40 hours of formal offsite instruction; (2) a minimum of 3 days of actual onsite field experience under the supervision of a trained and experienced field supervisor; and (3) 8 hours of annual refresher training. Field personnel who directly supervise employees engaged in hazardous waste operations also receive at least 8 additional hours of specialized supervisor training.

Copies of the field team's health and safety training records, including course completion certifications for the initial and refresher health and safety training, and specialized supervisor training are maintained in project files.

Before work begins at a specific hazardous waste project site, EA personnel are required to undergo site-specific training that thoroughly covers the following areas:

- Names of personnel and alternates responsible for health and safety at a hazardous waste project site
- Health and safety hazards present onsite
- Selection of the appropriate personal protective equipment (PPE)
- Correct use of PPE
- Work practices to minimize risks from hazards
- Safe use of engineering controls and equipment onsite
- Medical surveillance requirements, including recognition of symptoms and signs that might indicate overexposure to hazardous substances.

For more health and safety details, see EA's site-specific HASP, Revision 01 (EA 2016).

1.4.2 Subcontractor Training

Subcontractors who work on site will certify that their employees have been trained for work on hazardous waste project sites. Training will meet OSHA requirements defined in 29 Code of Federal Regulations 1910.120(e). Before work begins at the project site, subcontractors will submit copies of the training certification for each employee to EA.

All employees of associate and professional services firms and technical services subcontractors will attend a safety briefing and complete the Safety Meeting Sign-Off Sheet before they conduct onsite work. This briefing is conducted by the EA health and safety officer or other qualified person.

Subcontractors are responsible for conducting their own safety briefings. EA personnel may audit these briefings.

1.5 DOCUMENTATION AND RECORDS

The following sections discuss the requirements for documenting field activities and for preparing laboratory data packages. This section also describes reports that will be generated as a result of this project.

1.5.1 Field Documentation

Field personnel will use permanently bound field logbooks with sequentially numbered pages to record and document field activities and will follow Surface Water, Groundwater, and Soil/Sediment Field Logbooks – Revision 0, SOP 016 (Appendix A). The logbook will list the contract name and number; site name; and names of subcontractors, service client, and project manager. At a minimum, the following information will be recorded in the field logbook:

- Name and affiliation of all onsite personnel or visitors
- Weather conditions during the field activity
- Summary of daily activities and significant events
- Notes of conversations with coordinating officials
- References to other field logbooks or forms that contain specific information
- Discussions of problems encountered and their resolution
- Discussions of deviations from the SAP or other governing documents
- Description of all photographs taken.

The field team will also use the field forms included in Appendix B to record field activities.

1.5.2 Laboratory Documentation

This section describes the data reporting requirements for EA field personnel and laboratories (e.g., EPA CLP laboratory, EPA Region 6 Laboratory, or subcontractor non-CLP laboratory) that submit field and laboratory measurement data under the EPA Region 6 RAC program.

EA will require non-CLP laboratories to prepare and submit data packages in accordance with the EPA CLP protocols (EPA 2016) for hardcopy and EDD format of VOCs and SVOCs, if applicable. Data packages will include all appropriate documentation for independent validation of data and verification of the DQOs. The following documentation will be required for full data validation, if applicable:

- Case narratives, which will describe all QC non-conformances that are encountered during the analysis of samples in addition to any corrective actions that are taken
  - Statement of samples received
  - Description of any deviations from the specified analytical method
  - Explanations of data qualifiers that are applied to the data
  - Any other significant problems that were encountered during analysis.

- Tables that cross-reference field and laboratory sample numbers

- Chain-of-custody forms, which pertain to each sample delivery group or sample batch that is analyzed

- Laboratory reports, which must show traceability to the sample analyzed and must contain specified information
  - Project identification
  - Field sample number
  - Laboratory sample number
  - Sample matrix description
  - Dates and times of sample collection, receipt at the laboratory, preparation, and analysis
  - Description of analytical method and reference citation
  - Results of individual parameters, with concentration units, including second column results, second detector results, and other confirmatory results, where appropriate
  - Quantitation limits achieved
  - Dilution or concentration factors.

- Data summary forms and QC summary forms showing analytical results, if applicable
  - Samples
  - Surrogates
  - Blanks
  - Field QC samples
  - LCSs
  - Initial and continuing calibrations
  - Other QC samples.

- Laboratory control charts
  - Raw data
  - Instrument printouts

— Laboratory bench sheets for preparation of samples.

- MDL study results.

EA's project manager, in cooperation with the QA officer, will define site-specific requirements for data reporting. Requests for analytical services (discussed in Section 2.10) clearly define these requirements, the turnaround time for receipt of the data deliverables specified, and any requirements for retaining samples and laboratory records. Laboratory QA managers are responsible for ensuring that all laboratory data reporting requirements in the QAPP are met.

1.5.3 Level 4 Type Data Package

When a Level 4 type data package is required, the laboratory will prepare data packages in accordance with the instructions provided in the EPA CLP SOW (EPA 2016). These data packages will contain all of the information from the summary data package and all associated raw data. Data packages are due to EA within 35 days after the last sample in the sample delivery group is received. Unless otherwise requested, the subcontractor will deliver one copy of the final data package.

1.5.4 Reports Generated

Following the completion of the RA field program and receipt of validated data, EA will prepare the following reports associated with the site RA:

- Inspection Report
- Short Term and Long Term Monitoring Plan (per EPA's technical direction)
- Amended RA Report (as appendixes).

2. DATA GENERATION AND ACQUISITION

This section describes the requirements for the following:

- Sampling process design (Section 2.1)
- Project mobilizations (Section 2.2)
- Sampling methodology (Section 2.3)
- Sampling processing (Section 2.4)
- Decontamination (Section 2.5)
- Management of investigation-derived waste (Section 2.6)
- Sample designation (Section 2.7)
- Sample container, volume, preservation, and holding time requirements (Section 2.8)
- Sample handling and custody requirements (Section 2.9)
- Analytical methods requirements (Section 2.10)
- Quality control requirements (Section 2.11)
- Instrument calibration and frequency (Section 2.12)

- Requirements for inspection and acceptance of supplies and consumables (Section 2.13)
- Data acquisition requirements (Section 2.14)
- Data management (Section 2.15).

2.1 SAMPLING PROCESS DESIGN

In order to meet RA objectives, EA will implement the following field program to determine nature and extent of groundwater contamination in the MW-109A Area:

- Conduct a DPT investigation and collect groundwater samples from up to 20 DPT temporary well points to be analyzed using a field-portable gas chromatograph (GC)

- Install up to six new monitoring wells

- Collect groundwater samples during up to four groundwater sampling events from newly-installed and existing monitoring wells for analysis for VOCs and SVOCs.

Once the groundwater contamination is delineated, EA will use this information to support development of a Technical Impracticability Waiver Zone in the vicinity of the MW-109A Area.

EA will also perform a vapor intrusion assessment at a private residence in close proximity to the MW-109A Area to evaluate whether there is potential vapor intrusion risk to residents.

2.2 PROJECT MOBILIZATIONS

It is anticipated that up to four mobilizations will be required for activities associated with groundwater sampling and one mobilization will be required for the vapor intrusion air sampling. With EPA's approval, the EA project manager will identify the monitoring wells to be sampled in the MW-109A Area. Groundwater and air sampling methodologies to be implemented during each mobilization are discussed in Section 2.3.

Access agreements have been obtained by EPA and provided to EA for one residential and one industrial property. EA will attempt to notify the property owners prior to mobilization. Field staff collecting samples from the wells located on private property will maintain a current set of access agreements during all field activities. Signed copies of these records will be maintained in the project files.

2.3 SAMPLING METHODOLOGY

This section describes the procedures for sample collection, including sampling methods and equipment, sample preservation requirements, and decontamination procedures. Table 4 lists the SOPs that will be implemented during this field program. Copies of the SOPs are provided in Appendix A. Sample collection and handling procedures for samples that will be analyzed at the EPA CLP laboratory will follow CLP protocols in accordance with EPA's *Contract Laboratory Program Guidance for Field Samplers* (EPA 2014a). EA assumes that IDW streams may be

generated during the DPT investigation, monitoring well installation and development, and groundwater sampling activities.

2.3.1 Direct-Push Technology Groundwater Sampling

EA will install a series of laterally distributed temporary DPT borings for collection of up to 20 discrete groundwater samples along the predicted longitudinal and transverse axis of the expected plume downgradient of MW-109A.

EA will utilize a two-member field team (including one geologist) in addition to subcontractor personnel to conduct groundwater sampling activities using DPT. The soil will be logged by the geologist using the Unified Soil Classification System (USCS). Groundwater will be sampled according to the procedures outlined in EA SOP 047 (Appendix A). Groundwater samples will be obtained by advancing 1.5-inch-diameter nominal push rods with a screen point sampling tube. The rods will be advanced to a specified depth based on groundwater gauging data from nearby wells such that the screen point spans the water-bearing zone. Samples will then be collected either using a peristaltic pump (if possible) or an inertial pump (if depth to groundwater is too great for peristaltic pumping). Once the groundwater sample is obtained, the borehole will be grouted through the push rods upon retrieval. Groundwater samples will be analyzed for VOCs in the field using a portable GC (FROG 4000), so that real-time data are available for decision-making purposes. The plume delineation will proceed in a downgradient direction from MW-109A on 50-foot centers (to the degree possible given vegetation and accessibility) until the plume has been mapped in a downgradient sense. Once a clean groundwater sample has been obtained, an additional sample will be collected 25 feet upgradient of that point (i.e., between the last and second to last sample points) to halve the distance and refine the delineation. Once the downgradient delineation has been completed, a similar procedure will be employed to complete delineation to the east and west, and in the upgradient direction.

To the south of CR 126, transverse plume delineation will be completed at the approximate midpoint between MW-109A and the last downgradient DPT groundwater sample location that had detectable contamination. In this medial plume position, the width of the plume will be mapped, thereby completing horizontal plume delineation.

Vertical plume delineation will not be completed using DPT due to concerns over cross contamination. Rather, nested monitoring wells properly isolated from the shallow plume with conductor casing will be installed in the medial plume position along the longitudinal plume axis.

2.3.2 Monitoring Well Installation

EA anticipates installing and developing up to five new shallow monitoring wells: upgradient, cross-gradient west, cross-gradient east, downgradient, and one well in medial plume position (near the vertical well nest) along the plume centerline. One new deep monitoring well will be installed in the second water-bearing zone with the premise this well will be clean. These wells will be installed over a 6-day period using hollow-stem auger drilling methodology. Well locations and quantity will be based on data collected during the aforementioned DPT plume delineation activities and in collaboration with EPA. Soil cores will be logged by the geologist

using the USCS. The top-of-casing elevations for the new monitoring wells will be surveyed after completing installation activities as described in Section 2.3.4. Groundwater samples will be collected as described in Section 2.3.3.

2.3.3 Monitoring Well Sampling

This SAP is a living document with additional decisions regarding which wells will be sampled to be based on further analysis. The following monitoring wells may be sampled via HydraSleeve™ technology during the groundwater sampling events: MW-33, MW-108, MW-109A, MW-110, MW-191R, MW-192, and MW-193 (Figure 3). Monitoring well MW-191 was plugged and replaced by a new monitoring well, MW-191R, installed during the period-of-performance for this RA. The following groundwater samples will also be collected from up to six new monitoring wells installed under this RA: MW-201, MW-202, MW-203, MW-204 (Figure 3). EA will coordinate with EPA prior to each site mobilization to identify which wells are to be sampled and to ensure that the necessary access agreements are in place.

Monitoring wells will be gauged with a water level meter or interface probe using EA SOP 010 (Appendix A). Total well depth will be measured during each gauging event to determine if sediment is accumulating in the well sump.

EA will utilize a two-member field team to conduct groundwater sampling activities, with samples collected using HydraSleeves™. The HydraSleeve™ is a no-purge (passive) grab sampling device used to collect groundwater samples directly from the screened interval of a well without having to purge the well prior to sample collection. The HydraSleeve™ causes no drawdown in the well (until the sample is withdrawn from the water column) and only minimal disturbance of the water column, because it has a very thin cross section and it displaces very little water (less than 100 milliliters) during deployment in the well. The HydraSleeve™ collects a sample from within the screen only, and it excludes water from any other part of the water column in the well through the use of a self-sealing check valve at the top of the sampler. It is a single-use (disposable) sampler that is not intended for reuse, so there are no decontamination requirements for the sampler itself. Water quality parameters, including pH, water temperature, dissolved oxygen, specific conductance, oxidation-reduction potential, and turbidity, will be collected at each monitoring well location using a water quality meter. The SOP for sampling groundwater using a HydraSleeve™ is included in Appendix A.

Table 5 specifies the required sample volume, container type, preservation technique, and holding time for each analysis that is to be conducted on the groundwater samples. Required containers, preservation techniques, and holding times for field QC samples, such as field duplicates, field blanks, trip blanks, and MS/MD and MS/MSD samples will be the same as for field samples but will require additional volumes as described in Table 5.

2.3.4 Land Surveying

During the field program, EA will survey the locations for newly-installed monitoring wells using portable Global Positioning System (GPS) equipment. EA may also re-survey the locations of existing monitoring wells in the MW-109A Area. Elevations of top-of-casing for each monitoring well will be measured and referenced to a relative benchmark using either

differential leveling, or Real Time Kinematic GPS. As an alternative, EA will subcontract a local State of Texas-Registered Professional Licensed Surveyor to perform survey activities.

2.3.5 Indoor Air Sampling

EA will investigate the vapor intrusion pathway by conducting sub-slab soil gas and indoor air sampling at the residence located in the vicinity of MW-109A. Up to six air samples (two indoor air, two sub-slab, one outdoor air, and one field duplicate) will be collected from the residence to perform a vapor intrusion assessment. Sub-slab soil gas and indoor air samples will be collected from occupied areas of the subject residence in accordance with the vapor intrusion sampling procedures and applicable SOPs (Appendix A). Sub-slab soil gas samples will be used to evaluate the potential concentration of vadose zone source material and to evaluate the attenuation of vapors from source material (impacted groundwater and vadose zone) into indoor air. Indoor air samples will also be used to evaluate the attenuation of vapors from source material to indoor air as well as provide an exposure point concentration for the building occupants.

Air samples will be analyzed by a subcontractor non-CLP laboratory in accordance with EPA Method TO-15 Selective Ion Monitoring (SIM) (EPA 1999).

2.4 SAMPLE PROCESSING

Samples for fixed laboratory analysis will be processed and packaged in accordance with the *Contract Laboratory Program Guidance for Field Samplers* (EPA 2014a) and/or SOP 004 (Appendix A), as applicable.

2.5 DECONTAMINATION

Re-usable field equipment utilized during the field program will be decontaminated prior to and after use in accordance with SOP 005 (Appendix A). Decontamination of field equipment will occur in buckets, plastic containers, or other similar containers with sealing lids, and the resulting fluid will be transferred to an appropriate container staged in the designated staging area. The decontamination water will be properly sampled and disposed of following local, state, and federal guidelines (Section 2.5).

2.6 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

EA will incorporate best management practices for green remediation as it relates to the management of IDW. IDW will be characterized and managed in accordance with local, state, and federal laws, as applicable. In addition, EA may construct an evaporation pond or may use storage tanks to treat the IDW water onsite.

IDW soil will be containerized, labeled, and temporarily stored at the designated staging area until profiled for acceptance at an EPA approved disposal facility (SOP 042, Appendix A). IDW soil samples will be submitted to an EA subcontractor laboratory for disposal characterization. Landfill Disposal Restrictions will dictate sample quantities and analysis.

Decontamination water generated during well installation, groundwater sampling, and equipment decontamination will be containerized, labeled, and temporarily stored at the designated staging area until profiled for acceptance at an approved disposal facility (SOP 042, Appendix A). IDW water samples will be submitted to an EA subcontractor laboratory for disposal characterization.

2.7 SAMPLE DESIGNATION

Each sampling location will be designated with a unique alphanumeric designation according to the following sample classifications:

- **Soil Gas or Air Sample Designation.** Soil gas and air sample designations will include two fields that are separated by a dash; for example: SG-01, IA-01, or OA-01.
  - The first field, "SG,""IA," or "OA," denotes that the sample is sub-slab soil gas, indoor air, or outdoor ambient air, respectively.
  - The second field represents a unique numeric identifier.
  - EA may opt to add additional fields to further identify the sample locations as appropriate and necessary.

- **Groundwater Sample Designation.**
  - Groundwater sample designation for permanent monitoring wells will utilize two fields that are separated by a dash; for example: MW-109A.
    - The two fields will utilize the monitoring well designation as presented below.
  - Groundwater sample designation for temporary monitoring points will utilize two fields that are separated by a dash; for example: GW-01.
    - The two fields will utilize DPT temporary groundwater monitoring point designation as presented below.
  - EA may opt to add an additional field to identify the sampling depth as appropriate and necessary.

- **New Well Designation.** Newly-constructed monitoring well designation will include two fields that are separated by a dash; for example: MW-200 or GW-01.
  - The alpha characters in the first field, identifies the type of groundwater monitoring point, where:
    - "MW" represents a permanent groundwater monitoring well
    - "GW" represents a temporary groundwater monitoring point installed using DPT
  - The second field, "200" or "01," represents the permanent or temporary groundwater monitoring point designation or identifier.
  - EA may opt to add additional fields to further identify the monitoring wells as appropriate and necessary.

- **Field Duplicate Sample Designation.** Field duplicate samples will be identified by adding a "Dup" to the end of the sample designations described above; for example, MW-109A-Dup.

- **Field, Trip, and Equipment Rinsate Blank Sample Designation**. Trip, field, and equipment rinsate blank samples will be identified sequentially beginning with TB-1, FB-1, and ER-1, respectively.

2.8 SAMPLE CONTAINER, VOLUME, PRESERVATION, AND HOLDING TIME REQUIREMENTS

Table 5 specifies the required sample volume, container type, preservation technique, and holding time for each analysis that is to be conducted during each phase of sampling.

Required containers, preservation techniques, and holding times for field QC samples, such as field duplicates, field blanks, trip blanks, and MS/MD and MS/MSD samples, will be the same as for the associated field samples.

2.9 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Each sample collected by the EA field team will be traceable from the point of collection through analysis and final disposition to ensure sample integrity. Sample integrity helps to ensure the legal defensibility of the analytical data and subsequent conclusions. Sample handling will follow CLP protocols as required in EPA's *Contract Laboratory Program Guidance for Field Samplers* (EPA 2014a).

The EA field team will use EPA's data management system known as "Scribe" to generate all chain-of-custody records in the field. Applicable copies of generated Scribe files will be delivered to EPA data management personnel as required by CLP protocols.

2.9.1 Sample Documentation

Documentation during sampling is essential to ensure proper sample identification. EA personnel will adhere to the following general guidelines for maintaining field documentation:

- Documentation will be completed in permanent ink.
- All entries will be legible.
- Errors will be corrected by crossing out with a single line and then dating and initialing the lineout.
- Any serialized documents will be maintained at EA and referenced in the site logbook.
- Unused portions of pages will be crossed out, and each page will be signed and dated.

The EA field representative is responsible for ensuring that sampling activities are properly documented.

2.9.2 Sample Labels

A sample label will be affixed to each sample container. The label will be completed with the following information written in indelible ink:

- Project name and location
- Sample identification number
- Date and time of sample collection
- Sample collector's initials
- Analysis required.

2.9.3 Chain-of-Custody

EA will use standard sample custody procedures to maintain and document sample integrity during collection, transportation, storage, and analysis. A sample will be considered to be in custody if one of the following statements applies:

- It is in a person's physical possession or view.
- It is in a secure area with restricted access.
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Chain-of-custody procedures provide an accurate written record that traces the possession of individual samples from the time of collection in the field to the time of acceptance at the laboratory. The chain-of-custody record will be used to document all samples collected and the analysis requested. Information that the field personnel will record on the chain-of-custody record includes:

- Project name and number
- Sampling location
- Name and signature of sampler
- Destination of samples (laboratory name)
- Sample identification number
- Date and time of collection
- Analysis requested
- Signatures of individuals involved in custody transfer, including the date and time of transfer

- Airbill number (if applicable)

- Project contact and phone number.

Unused lines on the chain-of-custody record will be crossed out. Field personnel will sign chain-of-custody records that are initiated in the field, and the airbill number will be recorded. The record will be placed in a waterproof plastic bag and taped to the inside of the shipping container used to transport the samples. Signed airbills will serve as evidence of custody transfer between field personnel and the courier, and between the courier and the laboratory. Copies of the chain-of-custody record and the airbill will be retained and filed by field personnel before the containers are shipped.

The following procedures will be implemented when samples collected during this project are shipped to analytical laboratories (with shipping considerations applicable to laboratories other than geotechnical laboratories):

- The shipping box will be filled with bubble wrap, sample bottles, and packing material. Sufficient packing material will be used to prevent sample containers from breaking during shipment.

- The chain-of-custody records will be placed inside a plastic bag. The bag will be sealed and taped to the inside of the cooler lid. The airbill, if required, will be filled out before the samples are handed over to the carrier. The laboratory will be notified if the sampler suspects that the sample contains any substance that would require laboratory personnel to take safety precautions.

- The shipping box will be closed and taped shut with strapping tape around both ends.

- Signed and dated custody seals will be placed on the front and side of each shipping box. Wide clear tape will be placed over the seals to prevent accidental breakage.

- The chain-of-custody record will be transported within the taped sealed shipping box. When the shipping box is received at the analytical laboratory, laboratory personnel will open the shipping box and sign the chain-of-custody record to document transfer of samples.

2.10 ANALYTICAL METHODS REQUIREMENTS

The source of analytical services to be provided will be determined in part by DQOs and the intended use of the resulting data. EA will use CLP or EPA-approved methods for laboratory analyses of the samples.

EA will follow the analytical services request procedures that are outlined EA's Analytical Services Delivery Plan (EA 2005). If an analytical system fails, the QA officer will be notified, and corrective action will be taken. In general, corrective actions will include stopping the

analysis, examining instrument performance and sample preparation information, and determining the need to re-prepare and reanalyze the samples.

2.10.1 Field Analytical Methods

Water quality parameters, including pH, water temperature, dissolved oxygen, specific conductance, oxidation-reduction potential, and turbidity, will be collected at each monitoring well and DPT collection points using a water quality meter.

2.10.2 Laboratory Analytical Methods

Fixed-laboratory analyses of aqueous samples will be conducted by the EPA Region 6 Houston Laboratory or a designated CLP laboratory. All aqueous samples submitted to the analytical laboratory will be analyzed in accordance with CLP SOW SOM02.4 (EPA 2016). Exhibit C of the CLP SOW SOM02.4 lists the CRQLs, which are provided in Appendix C.

Soil gas and indoor air samples will be analyzed for VOCs by a non-CLP laboratory using EPA Method TO-15 SIM (EPA 1999). Appendix D presents the analytical reporting limits for Method TO-15 SIM.

IDW samples will be analyzed for waste disposal characterization parameters by a non-CLP laboratory. IDW water will be analyzed for VOCs and SVOCs. IDW soil will be analyzed for toxicity characteristic leaching procedure (TCLP) VOCs, TCLP SVOCs, TCLP metals, corrosivity (pH), total cyanide, total sulfide, and ignitability. Analytical methods are identified on Table 5.

2.11 QUALITY CONTROL REQUIREMENTS

Various field and laboratory QC samples and measurements will be used to verify that analytical data meet the QA objectives. Field QC samples and measurements will be collected to assess the influence of sampling activities and measurements on data quality. Similarly, laboratory QC samples will be used to assess how the laboratory's analytical program influences data quality. This section describes the QC samples that are to be analyzed during the site sampling activities for: (1) each field and laboratory environmental measurement method; and (2) each sample matrix type. Table 3 shows the acceptance criteria for each type of QC sample and Table 6 presents the frequency of QC samples to be collected at the Site.

2.11.1 Field Quality Control Requirements

Field QC samples will be collected and analyzed to assess the quality of data that are generated by sampling activities. These samples will include laboratory QC samples collected in the field, field duplicates, equipment rinsates, and MS/MSDs. QC samples collected in the field for fixed-laboratory analysis are presented in Table 6.

Field duplicates are independent samples that are collected as close as possible, in space and time, to the original investigative sample. Field duplicates can measure the influence of sampling and field procedures on the precision of an environmental measurement. They can also

provide information on the heterogeneity of a sampling location. Field duplicates will be collected at a frequency of one for every 10 aqueous and air samples, as listed in Table 6, or one per 20 field samples if directed by EPA. Immediately following or in conjunction with collection of the original sample, the field duplicates are collected using the same collection method.

Field blanks (for aqueous samples only) are collected to assess: (1) cross-contamination during sample collection, preservation, and shipment, and (2) cleanliness of the sample containers and preservatives. Field blank samples consist of sample containers filled with analytically-certified, organic-free water. One field blank sample will be collected for each day of groundwater sampling activities (specifically for VOC analysis). If any contaminant is present in the blank samples above the method detection limit, the result for associated field samples that contain the same contaminant will be qualified as potentially not detected if the concentration of the field sample is less than five times the concentration found in the blank.

Equipment rinsate blanks are collected when non-dedicated or non-disposable sampling equipment is used to collect aqueous samples and put the samples into containers. These blanks assess the cleanliness of the sampling equipment and the effectiveness of equipment decontamination. Equipment rinsate blanks are collected by pouring analyte-free water over the decontaminated surfaces of sampling equipment that contacts sampling media. Equipment rinsate blanks are collected after sampling equipment has been decontaminated, but before the equipment is reused for sampling. Equipment rinsate blanks will be collected (for aqueous samples only) if non-dedicated or non-disposable equipment is used. The equipment rinsate blanks will be collected at a frequency as listed in Table 6.

MS/MSD samples are laboratory QC samples that are collected for organic methods. For aqueous samples, MS/MSDs require double or triple the normal sample volume, depending on analytical laboratory specifications. Each MS and MSD sample is one sample, usually collected from one location at double the normal sample volume. In the laboratory, MS/MSDs are split and spiked with known amounts of target analytes. Analytical results for MS/MSDs are used to measure the precision and accuracy of the laboratory's analytical methods. Each of these QC samples will be collected and analyzed at a frequency of one for every 10 (or 20 depending on EPA directive) investigative aqueous samples per matrix when samples are submitted to the EPA Region 6 laboratory, or a frequency of one for every 20 investigative samples for CLP laboratories.

Temperature blanks are containers of deionized or distilled water that are placed in each cooler shipped to the laboratory. Their purpose is to provide a container to test the temperature of the aqueous samples in the respective cooler upon receipt at the laboratory.

Trip blanks are analyzed for VOCs only. VOC samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-lined septum of the sample vial; therefore, a VOC trip blank will be analyzed to monitor for possible sample contamination. The trip blank also screens for possible contamination of VOC samples during handling and shipment from the field to the laboratory. The trip blanks will be provided by the laboratory and placed in each cooler that contains VOC aqueous samples.

2.11.2 Laboratory Quality Control Requirements

All laboratories that perform analytical work under this project must adhere to a QA program that is used to monitor and control all laboratory QC activities. Each laboratory must have a written QA manual that describes the QA program in detail. The laboratory QA manager is responsible for ensuring that all laboratory internal QC checks are conducted in accordance with EPA methods and protocols, the laboratory's QA manual, and the requirements of this SAP.

Many of the laboratory QC procedures and requirements are described in EPA-approved analytical methods, laboratory method SOPs, and method guidance documents.

The CLP and EPA methods specify the preparation and analysis of QC samples, and may include, but are not limited to, the following types: (1) LCSs, (2) method blanks, (3) MS and MSD samples (organics), (4) surrogate spikes (organics), and (5) standard reference materials or independent check standards. The following subsections discuss the QC checks that will be required for this project. A copy of CLP SOW SOM02.4 Exhibit C (i.e., Organic Target Analyte List and CRQLs) is provided in Appendix C. Appendix D presents the analytical reporting limits for EPA Method TO-15 SIM.

2.11.2.1 Laboratory Control Sample

LCSs are thoroughly characterized, laboratory-generated samples that are used to monitor the laboratory's day-to-day performance of analytical methods. The results of LCS analyses are compared to well-defined laboratory control limits to determine whether the laboratory system is in control for the particular method. If the system is not in control, corrective action will be implemented. Appropriate corrective actions will include: (1) stopping the analysis; (2) examining instrument performance or sample preparation and analysis information; and (3) determining whether samples should be re-prepared or reanalyzed.

2.11.2.2 Method Blanks

Method blanks, which are also known as preparation blanks, are analyzed to assess the level of background interference or contamination in the analytical system and the level that may lead to elevated concentration levels or false-positive data. Method blanks will be required for all laboratory analyses and will be prepared and analyzed at a frequency of one method blank per every 20 samples or one method blank per batch, if the batches consist of fewer than 20 samples.

A method blank consists of reagents that are specific to the analytical method and are carried through every aspect of the analytical procedure, including sample preparation, cleanup, and analysis. The results of the method blank analysis will be evaluated in conjunction with other QC information to determine the acceptability of the data generated for that batch of samples. Ideally, the concentration of a target analyte in the method blank will be below the reporting limit for that analyte. For some common laboratory contaminants, a higher concentration may be allowed.

If the method blank for any analysis is beyond control limits, the source of contamination must be investigated, and appropriate corrective action must be taken and documented. This

investigation includes an evaluation of the data to determine the extent of the contamination and its effect on sampling results. If a method blank is within control limits but analysis indicates a concentration of analytes that is above the reporting limit, an investigation should be conducted to determine whether any corrective action could eliminate an ongoing source of target analytes.

For organic and inorganic analyses, the concentration of target analytes in the method blank must be below the reporting limit for that analyte for the blank to be considered acceptable. An exception may be made for common laboratory contaminants (e.g., methylene chloride, acetone, 2-butanone, and phthalate esters) that may be present in the blank at up to five times the reporting limit. These compounds are frequently detected at low levels in method blanks from materials that are used to collect, prepare, and analyze samples for organic parameters.

2.11.2.3 Matrix Spikes and Matrix Spike Duplicates

MSs and MSDs are aliquots of an environmental sample to which known concentrations of target analytes and compounds have been added. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis. If there are many target analytes, they will be divided into two to three spike standard solutions. Each spike standard solution will be used alternately. The MS, in addition to an unspiked aliquot, will be taken through the entire analytical procedure, and the recovery of the analytes will be calculated. Results will be expressed in terms of percent recoveries and RPD. The percent recoveries of the target analytes and compounds are calculated and used to determine the effects of the matrix on the precision and accuracy of the method. The RPD between the MS and MSD results is used to evaluate method precision.

The MS/MSD is divided into three separate aliquots, two of which are spiked with known concentrations of target analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately, and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results will be expressed as RPD and percent recovery and compared to control limits that have been established for each analyte. If results fall outside control limits, corrective action will be performed.

2.11.2.4 Surrogate Spikes

Surrogates are organic compounds that are similar to the analytes of interest in chemical properties but are not normally found in environmental samples. Surrogates are added to field and QC samples, before the samples are extracted, to assess the efficiency of the extraction procedure and to assess the bias that is introduced by the sample matrix. Results are reported in terms of percent recovery. Individual analytical methods may require sample reanalysis based on surrogate criteria.

The laboratory will use surrogate recoveries mainly to assess matrix effects on sample analysis. Obvious problems with sample preparation and analysis (such as evaporation to dryness or a leaking septum) that can lead to poor surrogate spike recoveries must be eliminated before low surrogate recoveries can be attributed to matrix effects.

2.11.3 Common Data Quality Indicators

This section describes how QA objectives for precision, accuracy, completeness, and sensitivity are measured, calculated, and reported.

2.11.3.1 Precision

Precision of many analyses is assessed by comparing analytical results of MS and MSD sample pairs for organic analyses, field duplicate samples, laboratory duplicate samples, and field replicate measurements. If precision is calculated from two measurements, it is normally measured as RPD. If precision is calculated from three or more replicates, relative standard deviation is calculated.

2.11.3.2 Accuracy

The accuracy of many analytical methods is assessed by using the results of MS and MSD samples for organic analyses, surrogate spike samples, LCSs, standard reference materials, independent check standards, and measurements of instrument responses against zero and span gases.

For measurements in which spikes are used, percent recovery will be calculated.

2.11.3.3 Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability are exceeded.

When all data validation is completed, the percent completeness value will be calculated by dividing the number of useable results by the total number of sample results planned for this investigation.

Completeness will also be evaluated as part of the data quality assessment process (EPA 2000c). This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected.

2.11.3.4 Sensitivity

The achievement of MDLs depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality and to ensure that analyses meet the QA objectives that have been established for sensitivity.

2.11.4 Instrument and Equipment Testing, Inspection, and Maintenance Requirements

This section outlines testing, inspection, and maintenance procedures for field equipment and instruments and for laboratory instruments.

2.11.4.1 General Requirements

Testing, inspection, and maintenance methods and frequency will be based on: (1) the type of instrument; (2) the instrument's stability characteristics; (3) the required accuracy, sensitivity, and precision of the instrument; (4) the instrument's intended use, considering project-specific DQOs; (5) manufacturer's recommendations; and (6) other conditions that affect measurement or operational control. For most instruments, preventive maintenance is performed in accordance with procedures and schedules recommended in: (1) the instrument manufacturer's literature or operating manual or (2) SOPs associated with particular applications of the instrument.

In some cases, testing, inspection, and maintenance procedures and schedules will differ from the manufacturer's specifications or SOPs. This can occur when a field instrument is used to make critical measurements or when the analytical methods that are associated with a laboratory instrument require more frequent testing, inspection, and maintenance.

2.11.4.2 Field Equipment and Instruments

The use of leased field equipment and instruments is anticipated to conduct groundwater sampling activities, which may include a peristaltic or inertial pump, water quality meter, FROG field GC, survey equipment/GPS, and/or a photoionization detection meter. In the case where leased field equipment is used, the vendor will be responsible for thoroughly checking and calibrating field equipment and instruments before they are shipped or transported to the field. Copies of testing, inspection, and maintenance procedures will be shipped to the field with the equipment and instruments.

After the field equipment and instruments arrive in the field, they will be inspected for damage. Damaged equipment and instruments will be replaced or repaired immediately. Battery-operated equipment will be checked to ensure full operating capacity; if needed, batteries will be recharged or replaced.

Following use, field equipment will be decontaminated properly before being returned to the source. When the equipment is returned, copies of any field notes regarding equipment problems will be included so that problems are not overlooked and any necessary equipment repairs are performed.

2.11.4.3 Laboratory Instruments

All laboratories that analyze samples collected under the EPA Region 6 RAC 2 program must have a preventive maintenance program that addresses: (1) testing, inspection, and maintenance procedures; and (2) the maintenance schedule for each measurement system and required support activity. This program is usually documented by a SOP for each analytical instrument that is to be used. Typically, the program will be laboratory-specific; however, it should follow requirements outlined in EPA-approved guidelines. Some of the basic requirements and components of such a program are as follows:

- As a part of its QA/QC program, each laboratory will conduct a routine preventive maintenance program to minimize instrument failure and other system malfunction.

- An internal group of qualified personnel will maintain and repair instruments, equipment, tools, and gauges. Alternatively, manufacturers' representatives may provide scheduled instrument maintenance and emergency repair under a repair and maintenance contract.
- The laboratory will perform instrument maintenance on a regularly scheduled basis. The scheduled service of critical items should minimize the downtime of the measurement system. The laboratory will prepare a list of critical spare parts for each instrument. The laboratory will request the spare parts from the manufacturer and will store the parts.
- Testing, inspection, and maintenance procedures described in laboratory SOPs will be performed in accordance with manufacturer's specifications and the requirements of the specific analytical methods that are used.
- All maintenance and service must be documented in service logbooks (or the site-specific logbook) to provide a history of maintenance records. A separate service logbook should be kept for each instrument; however, due to the limited scope of this project, the service records will be maintained in the site-specific field log book. All maintenance records will be traceable to the specific instrument, equipment, tool, or gauge.
- The laboratory will maintain and file records that are produced as a result of tests, inspections, or maintenance of laboratory instruments. These records will be available for review by internal and external laboratory system audits that are conducted under the EPA Region 6 RAC 2 program.

2.12 INSTRUMENT CALIBRATION AND FREQUENCY

This section describes the procedures for maintaining the accuracy of field equipment and laboratory instruments that are used for field tests and laboratory analyses. The equipment and instruments should be calibrated before each use or, when not in use, on a scheduled periodic basis.

2.12.1 Field Equipment

EA will perform calibration of field equipment during the Site field activities specified herein. Calibration of the multi-parameter water quality meter will be conducted daily prior to sample collection activities. Should water quality readings appear to be questionable during sample collection, EA will recalibrate the field equipment as deemed necessary. The equipment calibration procedures described below will be followed.

Equipment will be maintained and calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of results are consistent with the manufacturer's specifications and with project-specific DQOs. Upon arrival of the field sampling and measurement equipment, EA field personnel will examine it to verify that it is in good working condition. The manufacturer's operating manual and instructions that accompany the equipment will be consulted to ensure that all calibration procedures are followed. Measuring and testing equipment may be calibrated either internally—by using in-house reference standards—or externally—by agencies, manufacturers, or commercial laboratories. Calibration records will contain a reference identifying the source of the procedure and, where feasible, the actual

procedure. Each piece of measuring and testing equipment will also be accompanied by an equipment use log. The equipment use log (which may be contained within the site-specific field log book) will be kept current and may contain the following information: (1) date of use; (2) times of use; (3) operating and assisting technicians; (4) calibration status; and (5) comments.

2.12.2 Laboratory Instruments

All laboratory equipment that is used to analyze samples collected under the EPA Region 6 RAC 2 program will be calibrated on the basis of written SOPs that are maintained by the laboratory. Calibration records (including the dates and times of calibration and the names of the personnel performing the calibration) will be filed at the location at which the analytical work was performed and maintained by the laboratory personnel who performed QC activities. The laboratory QA manager is responsible for ensuring that all laboratory instruments are calibrated in accordance with the requirements of this SAP.

The laboratories will follow the method-specific calibration procedures and requirements for laboratory measurements. Calibration procedures and requirements will also be provided, as appropriate, for laboratory support equipment, such as balances, mercury thermometers, pH meters, and other equipment that is used to take chemical and physical measurements.

2.13 REQUIREMENTS FOR INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The EA project manager is responsible for identifying the types and quantities of supplies and consumables that are needed for collecting the samples for this Task Order. The project manager is also responsible for determining acceptance criteria for these items. Supplies and consumables can be received at either an equipment distribution center or a Site. When supplies are received, the EA field personnel will sort the supplies according to vendor, check packing slips against purchase orders, and inspect the condition of all supplies before the supplies are accepted for use on a project. If the supplies do not meet the acceptance criteria, deficiencies will be noted on the packing slip and purchase order. In addition, a form will be completed describing the problem and circumstances, and noting the purchase order number of the item. Afterward, the item will be returned to the vendor for replacement or repair.

2.14 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

For this project, EA anticipates acquiring data from non-direct measurements such as databases, spreadsheets, and literature files.

2.15 DATA MANAGEMENT

Data for this project will be obtained from a combination of sources, including field measurements, the EPA Regional laboratory, CLP laboratory, and EA subcontractor laboratories. The data-gathering process requires a coordinated effort and will be conducted by project staff members in conjunction with all potential data producers. The data will be obtained from the analytical service provider, when appropriate, in the form of an EDD, in addition to the required hard copy analytical data package. Formal verification (or validation) of data will not be

required for the EPA Region 6 laboratory or CLP laboratory data packages, but will be used for reporting purposes as delivered. Data received from EA subcontractor laboratories will be reviewed and electronic data will be verified against the Level 4 type data package.

Data tracking is essential to ensure timely, cost-effective, and high-quality results. Data tracking begins with sample chain-of-custody. When the analytical service provider receives custody of the samples, the provider will send a sample acknowledgment to EA. The sample acknowledgment will confirm sample receipt, condition, and required analyses. The EPA tracking software (Scribe) will contain all pertinent information about each sample and can track the data at each phase of the process. The tracking software carries the data through completion of the data validation.

3. ASSESSMENT AND OVERSIGHT

This section describes the field and laboratory assessments that may be conducted during this project, the individuals responsible for conducting assessments, corrective actions that may be implemented in response to assessment results, and how quality-related issues will be reported to EA and EPA.

3.1 ASSESSMENT AND RESPONSE ACTIONS

Under the EPA Region 6 RAC 2 program, performance and system audits of field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the following:

- Performance and system audits
  - — Audit personnel
  - — Audit scope of work
  - — Audit frequencies
  - — Audit reports.

- Corrective action
  - — Sample collection and field measurements
  - — Laboratory analyses.

Nonconforming items and activities are those that do not meet the project requirements, procurement document criteria, and approved work procedures. Nonconformance may be detected and identified by the following personnel:

- Project personnel—During field operations, supervision of subcontractors, and field inspections

- Testing personnel—During preparation for and performance of tests, equipment calibration, and QC activities

- QA personnel—During the performance of audits, surveillance, and other QA activities.

Each nonconformance that affects quality will be documented by the person who identifies or originates the nonconformance. Documentation of nonconformance will include the following components:

- Description of nonconformance
- Identification of personnel who are responsible for correcting the nonconformance and, if verification is required, for verifying satisfactory resolution
- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted
- Proposed schedule for completing corrective action and the corrective action taken.

Non-conformance documentation will be made available to the project manager, QA manager, and subcontractor (e.g., non-CLP laboratories) management personnel, as appropriate.

The field personnel and QA personnel, as appropriate, are responsible for notifying the project manager and the QA manager of the nonconformance. In addition, the project manager and the project staff, as appropriate, will be notified of significant non-conformances that could affect the results of the work. The project manager is responsible for determining whether notification of EPA is required.

The completion of corrective actions for significant non-conformances will be documented by QA personnel during future auditing activities. Any significant recurring nonconformance will be evaluated by project and QA personnel, as appropriate, to determine its cause. Appropriate changes will be instituted, under corporate or project procedures, to prevent recurrence. When such an evaluation is performed, the results will be documented.

3.2 REPORTS TO MANAGEMENT

Effective management of environmental data collection operations requires timely assessment and review of measurement activities. It is essential that open communication, interaction, and feedback be maintained among all project participants, including: (1) the EA QA manager, program manager, project manager, and technical staff; and (2) the EPA Region 6 Task Order Monitor and QA officer. EA prepares monthly progress reports for each Task Order that is conducted under the EPA Region 6 RAC 2 program. These reports address any QA issues that are specific to the Task Order and facilitate timely communication of such issues.

At the program level, the QA manager prepares quarterly status reports of QA issues that are related to EA's work on the EPA Region 6 RAC 2 program. These reports are distributed to EA's president, corporate QA manager, RAC 2 program manager, and, upon request, the EPA Region 6 project officer. QA status reports address the following areas:

- Results of QA audits and other inspections, including any quality improvement opportunities that have been identified for further action
- Instrument, equipment, or procedural problems that affect QA
- Subcontractor performance issues
- Corrective actions
- Status of previously reported activities and continuous quality improvement initiatives
- Work planned for the next reporting period.

4. DATA VALIDATION AND USABILITY

This section describes the procedures that are planned to review, verify, and validate field and laboratory data. This section also discussed procedures for verifying that the data are sufficient to meet DQOs and measurement quality objectives for the project.

4.1 DATA REVIEW AND REDUCTION REQUIREMENTS

This section focuses on data review and reduction requirements for work conducted under the EPA Region 6 RAC 2 program. Section 4.2 addresses data validation and verification requirements. Section 4.3 addresses reconciliation with DQOs.

Data reduction and review are essential functions for preparing data that can be used effectively to support project decisions and DQOs. These functions must be performed accurately and in accordance with EPA-approved procedures and techniques. Data reduction includes all computations and data manipulations that produce the final results that are used during the investigation. Data review includes all procedures that field or laboratory personnel conduct to ensure that measurement results are correct and acceptable in accordance with the QA objectives that are stated in this SAP. Field and laboratory measurement data reduction and review procedures and requirements are specified in previously discussed field and laboratory methods, SOPs, and guidance documents.

Field personnel will record, in a field logbook and/or on the appropriate field form, all raw data from chemical and physical field measurements. The EA field staff has the primary responsibility for: (1) verifying that field measurements were made correctly; (2) confirming that sample collection and handling procedures specified in this project-specific SAP were followed; and (3) ensuring that all field data reduction and review procedures requirements are followed. The EA field staff is also responsible for assessing preliminary data quality and for advising the data user of any potential QA/QC problems with field data. If field data are used in a project report, data reduction methods will be fully documented in the report.

The EPA Region 6 Houston Laboratory and/or CLP laboratory will complete data reduction for chemical and physical laboratory measurements and will complete an in-house review of all laboratory analytical results. The laboratory QA manager will be responsible for ensuring that all laboratory data reduction and review procedures follow the requirements that are stated in this

SAP. The laboratory QA manager will also be responsible for assessing data quality and for advising the EA QA manager of possible QA/QC problems with laboratory data.

4.2 VALIDATION AND VERIFICATION METHODS

All data that are used to support activities under the EPA Region 6 RAC 2 program must be valid for their intended purposes. This section outlines the basic data validation procedures that will be followed for all field and laboratory measurements. The following subsections identify personnel who are responsible for data validation and the general data validation process and EPA data validation guidance that will be followed.

4.2.1 Data Validation Responsibilities

When analytical services are provided by laboratories subcontracted by EA, EA is responsible for data validation. The QA Manager has primary responsibility for coordinating EA's data validation activities. EA will conduct full validation on 20 percent of all subcontractor non-CLP laboratory data for investigation samples. A data review will be performed on 100 percent of the data. Data validation and review will be completed by the project chemist or designee. When data is generated by the EPA Region 6 Laboratory in Houston, Texas, it will be used as received from the laboratory, with no further validation. Data from CLP laboratories are validated by EPA's Environmental Services Assistance Team and used as received.

4.2.2 Data Validation Procedures

The validity of a data set is determined by comparing the data with a predetermined set of QC limits. EA data reviewers will conduct a systematic review of the data for compliance with established QC limits (such as sensitivity, precision, and accuracy), on the basis of spike, duplicate, and blank sampling results that are provided by the laboratory. The data review will identify any out-of-control data points or omissions. EA data reviewers will evaluate laboratory data for compliance with the following information:

- Method and project-specific analytical service requests
- Holding times
- Initial and continuing calibration acceptance criteria
- Field, trip, and method blank acceptance criteria
- Surrogate recovery
- Field duplicates and MS and MSD acceptance criteria
- LCS accuracy

- Other laboratory QC criteria specified by the method or on the project-specific analytical service request form

- Compound identification and quantitation

- Overall assessment of data, in accordance with project-specific objectives.

EA will follow the most current EPA CLP guidelines (EPA 2014a, 2014b) for completing data validation for all applicable test methods. General procedures in the CLP guidelines will be modified, as necessary, to fit the specific analytical method that is used to produce the data. In all cases, data validation requirements will depend on: (1) DQO levels that are defined in Section 1.3; (2) reporting requirements that are defined in Section 1.5; and (3) data deliverables that are requested from the laboratory, as discussed in Section 1.5.

4.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The main purpose of a QA system is to define a process for collecting data that are of known quality, are scientifically valid, are legally defensible, and fully support any decisions that will be based on the data. To achieve this purpose, the SAP requires that DQOs be fully defined (Section 1.3). All other parts of the QA system must then be planned and implemented in a manner that is consistent with the DQOs. QA system components that follow directly from the DQOs include: (1) documentation and reporting requirements (Section 1.5); (2) sample process design and sampling methods requirements (Sections 2.1 and 2.3); (3) analytical methods and analytical service requests (Section 2.10); (4) QC requirements (Section 2.11); and (5) data reduction and validation and reporting methods (Section 4.1).

After environmental data have been collected, reviewed, and validated, the data will undergo a final evaluation to determine whether the DQOs specified in this SAP have been met. EA will follow EPA's data quality assessment (DQA) process to verify that the type, quality, and quantity of data that are collected are appropriate for their intended use (EPA 2006a, 2006b, 2006c).

The DQA process involves: (1) verifying that the data have met the assumptions under which the data collection design and DQOs were developed; (2) taking appropriate corrective action if the assumptions have not been met; and (3) evaluating the extent to which the data support the decision that must be made so that scientifically valid and meaningful conclusions can be drawn from the data. To the extent possible, EA will follow DQA methods and procedures that have been outlined by EPA (2006b, 2006c).

When the five-step data quality assessment process is not completely followed because the DQOs are qualitative, EA will systematically assess data quality and data usability. This assessment will include:

- A review of the sampling design and sampling methods to verify that these were implemented as planned and are adequate to support project objectives.

- A review of project-specific data quality indicators for PARCCS to determine whether acceptance criteria have been met.

- A review of project-specific DQOs to determine whether they have been achieved by the data collected.

- An evaluation of any limitations associated with the decisions to be made based on the data collected. For example, if data completeness is only 90 percent compared to a project-specific completeness objective of 95 percent, the data may still be usable to support a decision, but at a lower level of confidence.

Following the conclusion of the sampling events, the data evaluation will include:

- Data usability evaluations and field QA/QC.

- Data reduction and tabulation of the sample data. The data tables will include water quality parameters; summary of analytes in the groundwater for the specific sampling event; and summary of field quality control data.

- Attachments to the technical memorandum will include the groundwater sampling forms and the data generated from the EPA Region 6 Houston Laboratory, CLP laboratory or EA subcontractor laboratory.

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Figures

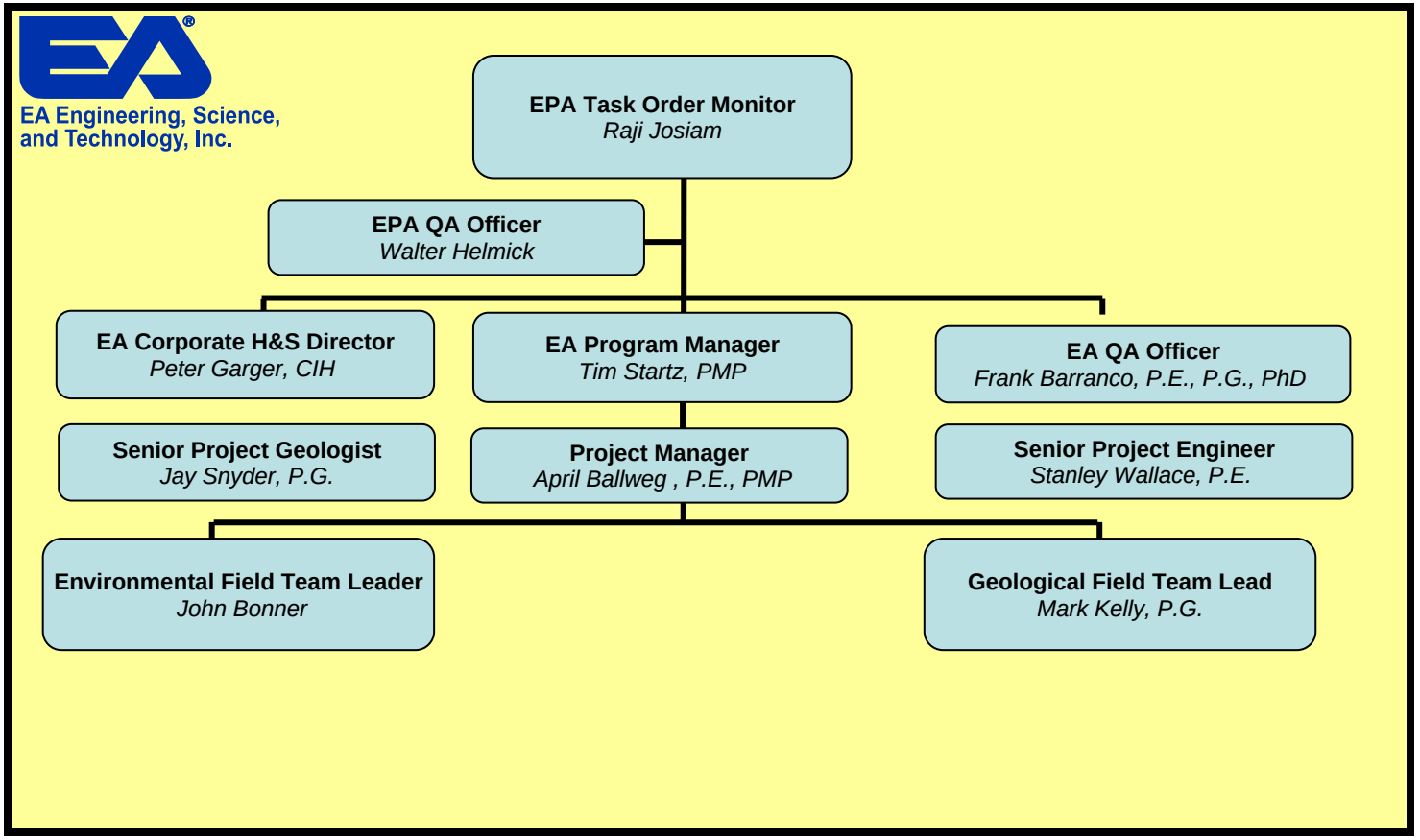
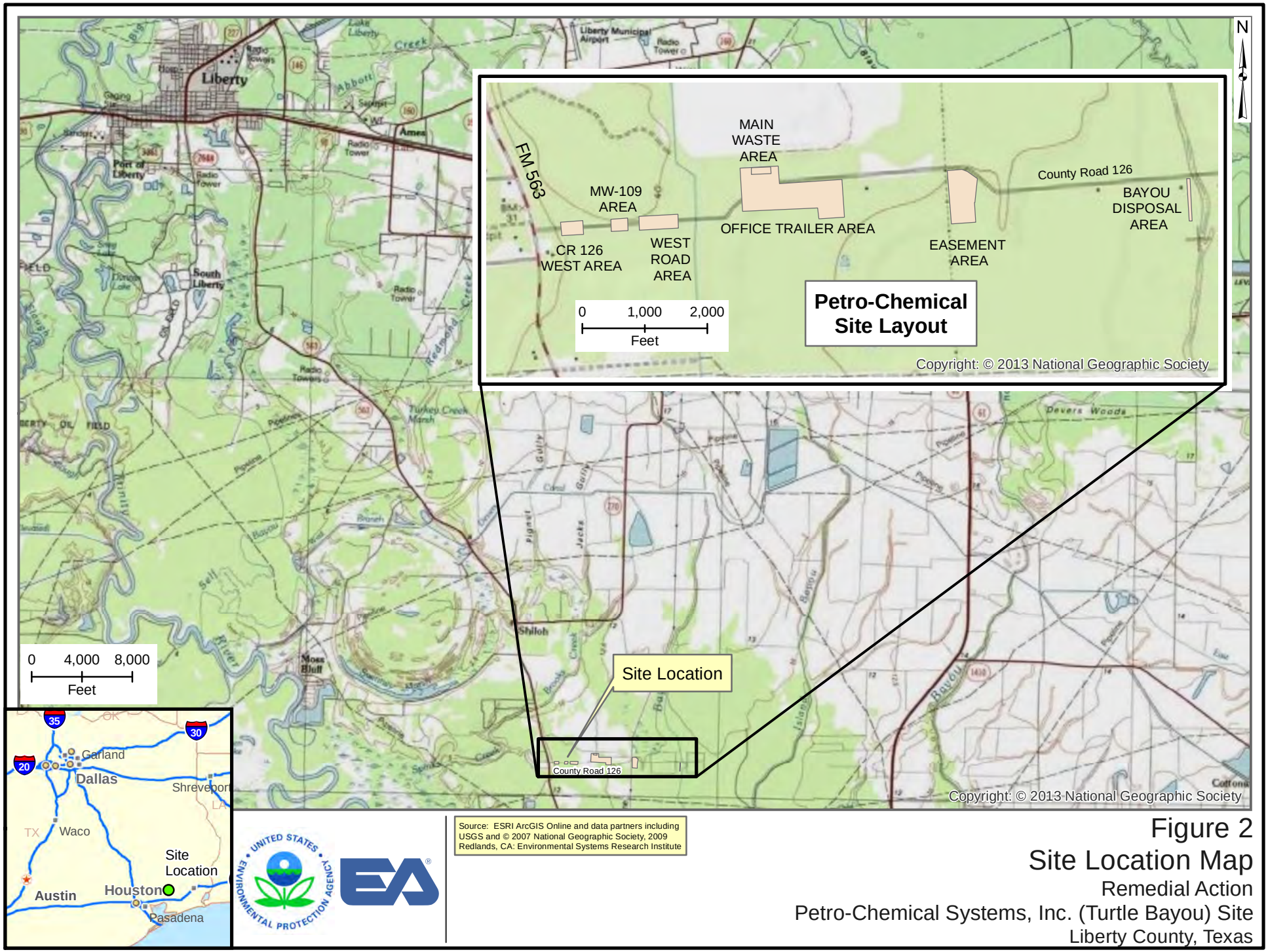


Figure 1. Project Organization.



Source: ESRI ArcGIS Online and data partners including USGS and © 2007 National Geographic Society, 2009 Redlands, CA: Environmental Systems Research Institute

Figure 2
Site Location Map
Remedial Action
Petro-Chemical Systems, Inc. (Turtle Bayou) Site
Liberty County, Texas

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Legend:

- Ground Water Monitoring Well
- Ground Water Monitoring Well - Plugged and Abandoned

0 25 50
Feet
1 inch = 50 feet
Source: Google Earth Pro, 2018.

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Figure 3
Monitoring Well Location Map
Remedial Action
Petro-Chemical System's Inc. (Turtle Bayou) Site
Liberty County, Texas

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Tables

TABLE 1 ELEMENTS OF EPA QA/R-5 IN RELATION TO THIS SAP

EPA QA/R-5 QAPP Element	EA SAP
A1 Title and Approval Sheet	Title and Approval Sheet
A2 Table of Contents	Table of Contents
A3 Distribution List	Distribution List
A4 Project/Task Organization	1.0 Project Description and Management
A5 Problem Definition/Background	1.1 Site Background and Problem Definition
A6 Project/Task Description	1.2 Description of Project Objectives and Tasks
A7 Quality Objectives and Criteria	1.3 Data and Measurement Quality Objectives
A8 Special Training/Certification	1.4 Special Training and Certification
A9 Documents and Records	1.5 Documentation and Records
B1 Sampling Process Design	2.1 Sampling Process Design 2.2 Project Mobilizations
B2 Sampling Methods	2.3 Sampling Methodology
B3 Sample Handling and Custody	2.4 Sample Handling and Custody Requirements
B4 Analytical Methods	2.5 Analytical Methods Requirements
B5 Quality Control	2.6 Quality Control Requirements
B6 Instrument/Equipment Testing, Inspection, and Maintenance	2.6.4 Instrument and Equipment Testing, Inspection, and Maintenance Requirements
B7 Instrument/Equipment Calibration and Frequency	2.7 Instrument Calibration and Frequency
B8 Inspection/Acceptance of Supplies and Consumables	2.8 Requirements for Inspection and Acceptance of Supplies and Consumables
B9 Non-direct Measurements	2.9 Data Acquisition Requirements (Non-Direct Measurements)
B10 Data Management	2.10 Data Management
C1 Assessment and Response Actions	3.1 Assessment and Response Actions
C2 Reports to Management	3.2 Reports to Management
D1 Data Review, Verification, and Validation	4.1 Data Review and Reduction Requirements
D2 Validation and Verification Methods	4.2 Validation and Verification Methods
D3 Reconciliation with User Requirements	4.3 Reconciliation with Data Quality Objectives

TABLE 2 DATA QUALITY OBJECTIVES

STEP 1: STATE THE PROBLEM

- The extent of ground water contamination around monitoring well MW-109A area is unknown. Plume delineation is needed in order to support a Technical Impracticability waiver.
- Vapor intrusion assessment at a residence in close proximity to MW-109A at Petro-Chemical Systems Superfund Site (Site) is needed in order to evaluate the vapor intrusion pathway.

STEP 2: IDENTIFY THE GOALS FOR THE STUDY

- Ground water collected from the direct-push technology (DPT) collection points will be used to aid in plume delineation and to determine placement of additional monitoring wells.
- Ground water samples will be collected from monitoring wells to help delineate ground water contamination.
- Soil and water samples will be collected for disposal of investigation-derived waste (IDW).
- Up to six air samples (2 indoor air, 2 sub-slab or crawlspace, 1 outdoor air, and 1 field duplicate) will be collected from the residence in close proximity to MW-109A, and data will be used to perform a vapor intrusion assessment.

STEP 3: IDENTIFY INPUTS TO THE DECISIONS

- Fixed-laboratory analytical results for ground water samples collected from monitoring wells.
- Portable gas chromatograph (GC) field screening results of ground water collected from DPT collection points.
- Fixed-laboratory analytical results for air samples collected at the private residence in close proximity to MW-109A.

STEP 4: DEFINE STUDY BOUNDARIES

- DPT sampling locations may be limited by surface and subsurface soil characteristics (e.g., compaction, depth to ground water, and vegetation).
- For horizontal plume delineation, DPT samples will be collected upgradient, downgradient, to the east, and to the west of monitoring well MW-109A until a clean field-screened ground water sample is encountered in all four directions. Plume delineation will proceed in a downgradient manner from MW-109A based on 50-foot centers. Once a clean ground water sample has been obtained, a sample will be collected 25 feet upgradient of that point (i.e., between the last and second to last DPT sample points) to halve the distance and refine the delineation. Once the downgradient delineation has been completed, a similar procedure will be employed to complete delineation to the east and west, and in the upgradient direction. To the south of County Road 126, transverse plume delineation will be completed at the approximate midpoint between MW-109A and the last downgradient DPT ground water sample location that had detectable contamination. In this medial plume position, the width of the plume will be mapped, thereby completing horizontal plume delineation.
- Up to six new monitoring wells will be installed in the MW-109A Area. Up to five new shallow monitoring wells will be installed to a depth of 30 feet below ground surface (bgs): 1 upgradient, 1 cross-gradient west, 1 cross-gradient east, 1downgradient, and 1 medial (near the vertical well nest) along the plume centerline. One deep monitoring well will be installed under the premise that the deeper ground water-bearing zone is not impacted.
- Air sampling will be collected at a time and date acceptable to the owner of the private residence in close proximity to MW-109A. Air sampling, including set-up, will take approximately 3 days to complete.
- The U.S. Environmental Protection Agency (EPA) will obtain property access agreements for the sampling of ground water from Site monitoring wells and indoor air from the residence in close proximity to MW-109A.

STEP 5: DEVELOP THE ANALYTIC APPROACH

- Ground water samples collected from the DPT collection points will be analyzed for volatile organic compounds (VOCs) using a portable GC.
- Ground water samples will be analyzed for VOCs and semivolatile organic compounds (SVOCs) using EPA Contract Laboratory Program (CLP) Statement of Work (SOW) SOM02.4 (EPA 2016) or equivalent method.
- Ground water analytical results will be compared to the respective EPA Maximum Contaminant Levels (MCLs) and/or Regional Screening Levels (RSLs) to determine ground water contamination extent at the MW-109A Area.
- Soil gas and air samples will be analyzed for VOCs using EPA Method TO-15 and TO-15 Selective Ion Monitoring (SIM).

IDW will be analyzed for Toxicity Characteristic Leaching Procedure SVOCs, VOCs, and metals, as well as reactivity (total cyanide and sulfide), corrosivity (pH), and ignitibility, using EPA methods to determine disposal options.

STEP 6: SPECIFY THE PERFORMANCE OR ACCEPTANCE CRITERIA

- Ground water samples from the DPT collection points will be analyzed using the portable GC at detection limits sufficient to perform field screening and horizontal plume delineation.
- Ground water samples from monitoring wells will be analyzed in accordance with EPA CLP SOW methods or using EPA analytical methods that have been selected based on the method reporting limits below EPA MCLs and/or RSLs.
- Soil gas and air samples will be analyzed by EPA methods that have been selected based on the reporting limits capable of evaluating concentrations below RSLs.
- QA samples will be collected during each phase of sampling to evaluate sampling techniques and consistency.
- Analytical results will be evaluated within their own tolerance limits and compared to MCLs and/or RSLs.

STEP 7: DEVELOP THE PLAN FOR OBTAINING DATA

- Ground water samples will be collected from temporary DPT ground water sampling points.
- During each of up to two sampling events, ground water samples will be collected from up to 6 new monitoring wells and up to 7 existing monitoring wells in the MW-109A Area using HydraSleeve™ samplers.
- Up to six soil vapor/air samples (2 indoor air, 2 sub-slab soil vapor, 1 outdoor air, and 1 field duplicate) will be collected at the residence in close proximity to MW-109A using Summa® canisters.

TABLE 3 DATA QUALITY INDICATOR CRITERIA

Data Quality Indicator	Analytical Parameter	QC Check Sample	Acceptance Criteria
Accuracy (water) (Percent Recovery)	SVOCs VOCs	MS and MSD Blanks [a]	50 to 150 percent recovery Less than CRQL
Accuracy (air) (Percent Recovery)	VOCs	LCS Blanks	70 to 130 percent recovery Less than reporting limit
Precision (water) (Relative Percent Difference)	SVOCs VOCs	MS and MSD Field duplicates	30 percent RPD 50 percent RPD
Precision (air) (Relative Percent Difference)	VOCs	Lab duplicates Field duplicates	30 percent RPD 50 percent RPD
Sensitivity (Quantitation Limits)	SVOCs VOCs	MS, MD, and MSD Field duplicates	Not applicable
Completeness	The objective for data completeness is 90 percent.		
Representativeness	The sampling network and the field screening analytical methods for this Site are designed to provide data that are representative of Site conditions.		
Comparability	The use of standard published sampling and analytical methods, and the use of QC samples, will ensure data of known quality. These data can be compared to any other data of known quality.		

NOTES:

[a] May include method blanks, reagent blanks, instrument blanks, calibration blanks, and other blanks collected in the field (such as trip or field blanks)

CRQL = Contract-required Quantitation Limit
LCS = Laboratory control sample
MS = Matrix spike
MSD = Matrix spike duplicate
QC = Quality control
RPD = Relative percent difference
SVOC = Semivolatile organic compound
VOC = Volatile organic compound

TABLE 4 STANDARD OPERATING PROCEDURES

SOP Number	SOP Title
001	Sample Labels
002	Chain-of-Custody Form
003	Subsurface Utility Clearance
004	Sample Packing and Shipping
005	Field Decontamination
006	Summa Canister Sampling
010	Water Level and Well Depth Measurements
013	Collection of Monitoring Well Samples
016	Surface Water, Groundwater, and Soil/Sediment Field Logbooks
019	Monitoring Well Installation
039	Sample Preservation and Container Requirements
042	Disposal of Investigation-Derived Material
046	Aqueous Diffusion Samplers
047	Direct-Push Sampling
048	Low-Flow Sampling
---	Vapor Intrusion Sampling Procedures
---	Sampling Ground Water With a HydraSleeve™

NOTE:
SOP = Standard operating procedures

TABLE 5 REQUIRED SAMPLE VOLUME, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES FOR INVESTIGATIVE SAMPLES

Parameter	Method	Volume and Container	Preservatives	Holding Time [a]
Ground Water and Liquid Investigation-derived Waste				
SVOCs	CLP SOM02.4[b]	Two 1-liter amber glass bottles with Teflon™-lined caps	Store at < 6°C	Extract 7 days, analyze 40 days
VOCs	CLP SOM02.4[b]	Three 40-milliliter amber glass vials with Teflon™-lined septa caps	Hydrochloric acid to pH < 2; store at <6°C	14 days
Soil Gas and Air (Vapor Intrusion Sampling)				
VOCs	EPA TO-15 SIM	One 6-liter evacuated certified-clean Summa® canister	NA	As soon as possible not to exceed 30 days
Investigation-derived Waste (Soil)				
TCLP SVOCs	SW-846 1311/ 8270D	One 8-ounce glass jar	Store at < 6°C	14 days
TCLP VOCs	SW-846 1311/ SW8260C	One 8-ounce glass jar	Store at < 6°C	7 days extraction, 40 days analysis
TCLP metals	SW-846 1311/ 6010C/7470A	One 8-ounce glass jar	Store at < 6°C	180 days (28 days for mercury)
Corrosivity - pH	SW-846 9045D	One 4-ounce glass jar	Store at < 6°C	Analyze upon receipt
Total Cyanide (reactivity)	SW-846 9012B	One 8-ounce glass jar	Store at < 6°C	14 days
Total Sulfide (reactivity)	SW-846 9034	One 8-ounce glass jar	Store at < 6°C	7 days
Ignitability	SW1010A/1030	One 4-ounce glass jar	Store at < 6°C	NA

NOTES:

a Holding time is measured from the time of sample collection to the time of sample extraction and analysis.

b U.S. Environmental Protection Agency (EPA). 2016. *Multi-Media, Multi-Concentration, Contract Laboratory Program Statement of Work for Organics Analysis SOM02.4*. OSWER. Washington, D.C. October.

NA = Not applicable
SIM = Selective ion monitoring
SVOC = Semivolatile organic compound
TCLP = Toxicity characteristic leaching procedure
VOC = Volatile organic compound

TABLE 6 FREQUENCY OF FIELD QUALITY CONTROL SAMPLES

Field QC Sample	Frequency	
	Aqueous Samples	Air Samples
Field duplicate [a]	1 per 10 samples	1 per 10 samples
Field blank	1 per day (aqueous VOCs), if site conditions render this sample necessary	None
Equipment rinsate blank	1 per non-dedicated equipment type per day or 1 per 20 samples	Not applicable
MS/MSD [b] (organics)	1 per 20 samples (or per EPA Region 6 Laboratory requirements)	Not applicable
Temperature blank	1 per cooler	Not applicable
Trip blank	1 per cooler containing aqueous samples for VOC analysis	Not applicable

NOTES:

[a] CLP field duplicates may be collected at a frequency of 1 per 20 samples, if so directed by EPA.

[b] MS and MSD samples are technically not field QC samples; however, they require that the field personnel collect additional volume of sample materials and are, therefore, included on this table for reference. EPA may opt to exclude these extra volume requirements.

CLP = Contract Laboratory program
EPA = U.S. Environmental Protection Agency
MS = Matrix spike
MSD = Matrix spike duplicate
QC = Quality control
VOC = Volatile organic compound

Appendix A

Standard Operating Procedures

Standard Operating Procedure No. 001
for
Sample Labels

Prepared by

EA Engineering, Science, and Technology, Inc., PBC
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Hunt Valley, Maryland 21031

Revision 1
November 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 001	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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CONTENTS

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ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	29 November 2018	Systematic review and update	Dan Hinckley Sheena Styger Sanita Corum	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is provided below. Other formats with similar levels of detail are acceptable. Some project software including Scribe (U.S. Environmental Protection Agency (EPA)-associated projects) and FUDSchem (U.S. Army Corps of Engineers-associated projects) can generate pre-prepared labels thus minimizing efforts in the field.

NOTE: It is important to review with the Project/Program Manager to determine if client or project-specific modifications to this SOP are required. For example, if using EPA laboratories, case numbers may be assigned in lieu of having site or project names on the label.

PROJECT NAME _____ PROJECT NUM. _____
SAMPLE LOCATION/SITE ID _____
DATE: ____/____/____ TIME: _____:_____
ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER
FILTERED: [NO] [YES]
PRESERVATIVE: [NONE] [HNO_3] [OTHER _____]
SAMPLER: _____

2. MATERIALS

The following materials may be required:

- Sample label
- Indelible marker.

3. PROCEDURE

The following sections describe how to use the sample labeling system.

3.1 LABEL INFORMATION

As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

- Project name (do not include if there is a project or client-specific requirement to exclude)
- Project Number (or Case Number, as applicable)
- Location/site identification—enter the media type (i.e., well number, surface water, soil, etc.) sampling number, and other pertinent information concerning where the sample was taken
- Date of sample collection
- Time of sample collection
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with laboratory *prior to start of work)*
- Whether filtered or unfiltered (water samples only)
- Preservatives (water samples only)
- Number of containers for the sample (e.g., 1 of 2, 2 of 2).

3.2 ROUTINE CHECK

Double-check the label information to make sure it is correct. Detach the label, remove the backing, and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

3.3 RECORD INFORMATION

Record the sample number and designated sampling point in the field logbook, along with the following sample information:

- Time of sample collection (each logbook page should be dated)
- Location of the sample
- Organic vapor meter or photoionization meter readings for the sample (when appropriate)
- Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)
- Number of containers required for each sample

- Whether the sample is a quality assurance sample (split, duplicate, matrix spike/matrix spike duplicate, or blank).

3.3.1 Logbook Entry

A typical logbook entry might look like this:

- 7:35 a.m. Sample No. MW-3. Photoionization Detector = 35 parts per million.
- Petroleum odor present. Sample designated MW-3-001.

NOTE: Duplicate samples may be given a unique sample designation rather than the actual sample number with an added prefix or suffix. This will prevent any indication to the laboratory that this is a duplicate sample thus making it "blind" to the laboratory. This fictitious sample number must be listed in the logbook along with the actual location of the sample.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

If "blind" field duplicate samples have been called for, then no indication of which samples are duplicates is to be provided to the laboratory.

6. REFERENCES

Not applicable.

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Standard Operating Procedure No. 002
for
Chain-of-Custody Form

Prepared by

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Revision 1
November 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 002	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	29 November 2018	Systematic review and update	Dan Hinckley, Sheena Styger, Sanita Corum	Matthew Bowman

1. SCOPE AND APPLICATION

A chain-of-custody record (attached) is used as physical evidence of sample custody and as a permanent record for each sample collected. A chain-of-custody record documents the exchange and transportation of samples from the field to the laboratory. The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for use of the chain-of-custody form. Three example forms are provided as Figures SOP002-1 (EA's standard electronic chain-of-custody form), SOP002-2 (EA's Toxicology Laboratory chain-of-custody form), and SOP002-3 (U.S. Environmental Protection Agency [EPA] Scribe chain-of-custody form). Other formats with similar levels of detail are acceptable.

Most EPA projects utilize sampling and chain-of-custody instructions as documented in EPA's Samplers Guide (2014), which includes the use of Scribe, an in-house software program used to establish computer records of all environmental data and includes generation of chain-of-custodies. Using Scribe requires training, and the software and guidance can be found at the following link: https://response.epa.gov/site/site_profile.aspx?site_id=ScribeGIS. Training on Scribe is necessary and can be obtained through the Scribe weblink.

All new U.S. Army Corps of Engineers projects require the use of Formerly Used Defense Sites chemistry database (FUDSchem), which can be found at the following link: http://fudschem.com/public/framework/bannerhtml.aspx?dsn=systm&idhtml=10642&themesuffix=default&banner=banner_fudschem.jpg. This software will generate chain-of-custody forms specific to the sampling session. As with Scribe, FUDSchem training is necessary.

It is essential that chain-of-custody forms be completed properly, and that sample relinquishment be signed and dated appropriately. Laboratories use chain-of-custodies as their statement of work and, if it is not correct, the samples will not be analyzed appropriately. Sample custody documentation assures that the particular samples have been in secure locations, and that none of them have been tampered with, thus assuring appropriate results.

2. MATERIALS

The following materials may be required: chain-of-custody form and indelible ink pen.

3. PROCEDURE

- Give the site name and project name/number.
- Enter the sample identification code.
- Indicate the sampling dates for all samples.
- List the sampling times (military format) for all samples.

- Enter the total number of containers per cooler.
- List the analyses/container volume.
- Obtain the signature of sample team leader.
- State the carrier service and airbill number, analytical laboratory, and custody seal numbers (if applicable).
- Sign, date, and time the "relinquished by" section. Be sure the carrier signs and enters dates and time of acceptance of the samples.
- Upon completion of the form, retain a copy or portable document format, and affix the laboratory copy to the inside of the sample cooler in a zip-seal bag to protect from moisture, to be sent to the designated laboratory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency (EPA). 2014. Sampler's Guide, Contract Laboratory Program Guidance for Field Samplers. EPA/540/R014/013, Directive 92400.2-147. October.

Figures

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Figure SOP002-1 EA Chain-of-Custody Form

Company Name:

Project Manager or Contact:

Phone:

Project No.

Dept.: Task:

Project Name:

Sample Storage Location:

P.O. No.:

Page of

Report No.:

Parameters/Method Numbers for Analysis

Chain-of-Custody Record

EA Laboratories
231 Schilling Circle
Hunt Valley, MD 21031
Telephone: (410) 584-7000

Report Deliverables:
1 2 3 4 D E

EDD: Yes/No

DUE TO CLIENT: __________

Date	Time	Water	Soil	Sample Identification 19 Characters	**No. of Containers**														**EA Labs Accession Number**	Remarks
				XXXXXXXXXXXXXXXXXXX																**LPM**:
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
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Company Name:	Project Manager or Contact: Phone:	Parameters/Method Numbers for Analysis	Chain-of-Custody Record
Project No. Dept.: Task:	Project Name:		EA Laboratories 231 Schilling Circle Hunt Valley, MD 21031 Telephone: (410) 584-7000
Sample Storage Location:	P.O. No.:		**Report Deliverables:** **1 2 3 4 D E** **EDD: Yes/No** **DUE TO CLIENT:** _________
Page of	**Report No.:**		

Date	Time	Water	Soil	Sample Identification 19 Characters	**No. of Containers**														**EA Labs Accession Number**	Remarks
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																
				XXXXXXXXXXXXXXXXXXX																

Samples by: (Signature)	Date/Time |	Relinquished by: (Signature)	Date/Time |	Received by: (Signature)	Date/Time |
Relinquished by: (Signature)	Date/Time |	Received by Laboratory: (Signature)	Date/Time |	Airbill Number:	Sample Shipped by: (Circle) Fed Ex. Puro. UPS Hand Carried Other:

Cooler Temp. _______**C pH:** ___ **Yes** ___ **No Comments:** **Custody Seals Intact** ___ **Yes** ___ **No**

NOTE: Please indicate method number for analyses requested. This will help clarify any questions with laboratory techniques.

Figure SOP002-2 EA Toxicology Laboratory Chain-of-Custody Form

Client:	Project Manager: Phone: Project Contact: Phone:	No. of Containers																									
Project Name: Project#:																											
Page 1 of 1																											
Sample Collected		Matrix		SAMPLE IDENTIFICATION																							
Date	Time	Sediment	Water																								
Sampled by: (Signature)		Date/Time		Relinquished by: (Signature)																						Date/Time	
Relinquished by: (Signature)		Date/Time		Received by Laboratory: (Signature)																						Date/Time	

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Figure SOP002-3 EPA Scribe Chain-of-Custody Form

Page 1 of 1

USEPA
Lab Contact: John Smith
CarrierName: UPS
AirbillNo:
1Z2886820195095104

CHAIN OF CUSTODY RECORD
Site #: 47909
Contact Name: Michael Smith
Contact Phone: 800-332-0534

No: 5-112818-101859-0021
Cooler #: 13
Case #: 47909
Lab Phone: 800-660-1990

Lab #	Sample #	Location	Analyses	Matrix	Sample Date	Sample Time	Numb Cont	Container	Preservative	Lab QC
	HT18-01	HT18-01	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	
	HT18-02	HT18-02	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	
	HT18-03	HT18-03	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	

Special Instructions:	SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt

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Standard Operating Procedure No. 003
for
Subsurface Utility Clearance

Prepared by

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Revision 1
July 2018

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PROJECT-SPECIFIC VARIANCE FORM

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☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 003	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	6/28/2018	Systematic review and update	Matt Bowman	Pete Garger

1. SCOPE AND APPLICATION

1.1 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to prevent injury to workers and damage to subsurface structures (including tanks, pipe lines, water lines, gas lines, electrical service, etc.) during ground disturbance activities (including drilling, augering, sampling, use of direct-push technologies, excavation, trenching, concrete coring or removal, fence post installation, grading, or other similar subsurface operations).

1.2 LIMITATIONS

The procedures set forth in this document are general guidance, but may not be entirely applicable to particular sites based on the site-specific considerations. The Project Manager is responsible for making a site-specific evaluation of each site to determine how subsurface utility clearance procedures should be utilized or modified. If safety or other site-specific considerations require a modified or different procedure, the Project Manager should review the modified procedure with the Business Unit Director, Profit Center Manager, or Senior Technical Reviewer. Evaluation support of modified procedures may be provided by the Corporate Health and Safety Director or the Lead Construction Quality Engineer.

Special considerations may be required for utility location activities at complex or challenging project sites (underwater utilities, hazardous waste sites, etc.). Additional subsurface utility clearance procedures should be added as appropriate for difficult sites. When health and safety risks to workers or potential utility damage cannot be effectively managed through utility location, clearance, and protection measures, the Project Manager must consider the modification of ground disturbance activities (e.g., establishing a safe offset from high risk utilities). In these cases, detailed coordination with the client and/or regulatory staff is likely required.

1.3 SCOPE

This SOP provides minimum guidance for subsurface utility clearance activities, which must be followed prior to and during ground disturbance activities at EA project sites. Even after completing the subsurface utility clearance activities required in this SOP, all ground disturbance activities should proceed with due caution.

Deviations from this SOP may be provided on an exception basis for specific situations, such as underground storage tank systems removals, verified aboveground/overhead services/lines, undeveloped land/idle facilities, shallow groundwater conditions, soil stability, or well construction quality assurance/quality control concerns, etc.

EA or its subcontractors are responsible for, and shall ensure that, all ground disturbance activities are completed safely, without incident, and in accordance with applicable federal, state, and local regulations.

This SOP shall not override any site-specific or consultant/contractor procedures that are more stringent or provide a greater degree of safety or protection of health or the environment.

2. PROCEDURES

The EA Project Manager or his/her designee must complete the Subsurface Utility Clearance Checklist (Attachment A) in conjunction with the following procedures. The checklist must be completed before initiating any ground disturbance activities. The completed checklist must be submitted to the appropriate team individuals, subcontractors, and/or the client and included in the project files.

2.1 SAFETY

A Health and Safety Plan must be available onsite and followed by all contractors and subcontractors.

Work areas should be defined and secured with safety cones, safety tape, construction fence, other barriers, or signs as appropriate.

Site work permits must be obtained as required by site procedures. Based on site conditions or classification, the use of intrinsically-safe equipment may be required.

To ensure the safety of all onsite personnel and subsurface structure integrity, consideration should be given to de-energizing and locking out selected site utilities or temporarily shutting down a portion of or the entire facility.

2.2 SUBSURFACE UTILITY LOCATION ACTIVITIES

To gather all relevant information about potential subsurface structures prior to ground disturbance activities, the project team should pursue multiple lines of evidence on the type, location, depth, size, material of construction, and status (active/abandoned) of all utilities within and near the area planned for ground disturbance activities. A minimum of three lines of evidence should be obtained and documented; however, additional lines of evidences should be secured when possible. Lines of evidence may include the following:

- Historical Site Information
- Public Utility Mark-Out (One Call – 811)
- Private Utility Mark-Out
- Site Inspection
- Client/Facility Interviews and Coordination.

2.2.1 Historical Site Information

The most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) should be obtained, as available.

NOTE: As-built drawings may not accurately depict the locations and depths of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

EA should obtain any other site information such as easements, right-of-ways, historical plot plans, fire insurance plans, tank (dip) charts, previous site investigations, soil surveys, boring logs, and aerial photographs, etc. as relevant to the planned ground disturbance activities. Where applicable, EA should also contact contract personnel who may have historical site knowledge.

2.2.2 Public and Private Utility Mark-Outs

EA must ensure that a thorough mark-out at the site is completed to locate electrical, gas, telephone, water, sewer, low voltage electric lines, product delivery pipelines, fiber optic, and all other subsurface utilities/services.

- Where available, public utility companies must be contacted to identify subsurface utilities. (This can be accomplished through the One-Call system in most instances.) Attachment B provides a brochure for the 811 Utility Locate Call Center.

- In addition, where available and warranted by site conditions, a private utility/pipeline mark-out company should be contracted to perform an electronic subsurface survey to identify the presence of suspected hazardous or critical subsurface utilities and structures. In some cases, this is necessary to confirm public utility mark-outs in the vicinity of planned ground disturbance activities.

EA will review all available site plan subsurface information with the private mark-out company to assist in locating utilities and other subsurface structures.

NOTE: Mark-outs may not accurately depict the exact locations of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

Where possible, EA personnel are encouraged to be onsite at the time of subsurface mark-outs. This is to ensure accuracy and understanding of subsurface utility structures identified and provides an opportunity to exchange information with mark-out company personnel regarding planned work activities.

Subsurface utility structures should be marked throughout the entire work area(s) with adequate materials (e.g., site conditions may require paint and tape/flags). Ground disturbance activities must be started within 30 days of mark-out, unless local ordinances specify a shorter time period.

If activities are not started within required time period or markings have faded, mark-outs must be redone.

EA personnel will record time and date of mark-out request and list all companies contacted by the service and confirmation number. This information should be available for review onsite and checked off after visual confirmation of markings.

2.2.3 Site Inspection

To compare the site plan to actual conditions based on information gathered in other lines of evidence, a site inspection should be performed to identify potential signs of subsurface utilities. These signs may include:

- Signage identifying subsurface utilities
- Asphalt patching or paving scars
- Pull boxes, junction boxes, valve box covers, or manhole covers
- Sewer drains and clean-out traps
- Meters and light poles
- Piping or conduit on the walls or roofs of buildings
- Linear ground depressions
- Markings from previous utility mark-out efforts
- Other utilities including fire hydrants, on/below grade electrical transformers, splice cages, sprinkler systems, steam lines (including insulated tanks that may indicate steam lines), and cathodic protection on lines/tanks.

EA will document all findings and update the site plan with this information. In some regions, it may be more effective and efficient to conduct the site inspection at the same time the contractor performing the ground disturbance activity is mobilized to the site. The site inspection may include others as determined by the consultant/contractor and the Project Manager.

2.2.4 Client/Facility Interviews and Coordination

Knowledgeable client and facility staff familiar with site utilities should be interviewed to obtain information and documentation on potential subsurface utility locations, depth, etc. Results of these interviews should be documented and included with the Subsurface Utility Clearance Checklist. On third party sites, close coordination with the site owner's representatives for mark-outs, review of as-builts, and other information reviews should be conducted prior to any ground

disturbance work. Project Managers are encouraged to provide updated as-built information to the client.

EA will review the selected ground disturbance locations with the client. EA will not proceed with the subsurface activities until the plan has been discussed with the client. During execution of the project, if subsurface activities are required outside of the area previously approved by the client, EA will submit these changes to the client for approval prior to execution.

2.2.5 Ground Disturbance Activity Sequence

When practical, EA will plan ground disturbance activities starting at the point farthest from the location of suspected underground improvements. This is done to determine the natural subsurface conditions and to allow EA site personnel to recognize fill conditions.

Experience has shown that the following warning signs may indicate the presence of a subsurface structure:

- Warning tape (typically indicative of underground services).
- Pea gravel/sand/non-indigenous material (typically indicative of tanks or lines).
- Red concrete (typically indicative of electrical duct banks).
- The abrupt absence of soil recovery in a hand auger. This could indicate pea gravel or sand that has spilled out of the auger. This may not be indicative in areas where native soil conditions typically result in poor hand auger recoveries.
- Any unexpected departure from the native soil or backfill conditions as established by prior onsite digging.

If any of these conditions is encountered by EA site personnel, digging should stop and the client should be contacted.

3. UTILITY PROTECTION MEASURES DURING GROUND DISTURBANCE ACTIVITIES

After mobilization, but prior to the primary ground disturbance activities, the physical location of subsurface utilities should be cleared and verified whenever possible and practical. The clearance method used to clear and verify the subsurface utilities should be compatible with the inherent associated risk given the type of facility/property, subsurface utility material of construction, utility depth, soil stratigraphy, and the location of the ground disturbance activity, such that required delineation is obtained. It should be noted that in areas where there is paving, sufficient paving should be removed to allow clear visibility of the subsurface conditions during

clearance activities. The following is a list of potential clearance methods that may be used on a job site:

- Vacuum digging
- Probing
- Hand digging
- Hand augering
- Post-hole digging.

EA personnel will evaluate the potential for electrical shock or fire/explosion for each subsurface disturbance project and will evaluate as necessary the use of non-conductive or non-sparking tools (i.e., fiberglass hand shovels, and thick electrically insulating rubber grips on hand augers or probes). The potential need for the use of non-conductive materials, electrical safety insulated gloves, and footwear will also be evaluated on a case-by-case basis.

For drilling, direct-push technology, fence post installation, or other borehole installation, the area to be delineated will exceed the diameter of the largest tool to be advanced and sufficiently allow for visual inspection of any obstructions encountered.

3.1 SUBSURFACE CLEARANCE PROCEDURES FOR TRENCHING/ EXCAVATION ACTIVITIES

For trenching and excavation activities, appropriate subsurface clearance methods should be conducted along the length and width of the excavation at a frequency sufficient to ensure adequate precautions have been applied to the entire work area. The frequency and density of investigations will be based on site knowledge, potential hazards, and risks of the work area to surrounding locations.

Whenever subsurface structures are exposed, EA will cease work and mark the area (e.g., flags, stakes, cross bracing) to ensure the integrity of these exposed structures is maintained during subsequent trenching/excavation/backfilling.

During ground disturbance activities, EA and its subcontractors should consider the use of spotters to monitor the excavation for signs of subsurface utilities (pipes, conduits, cables, bedding material, warning tape, tracing wire, soil material changes, etc.) to provide early warning in the event unknown subsurface utilities are encountered. The decision to use spotters should be based on the risk of encountering unknown subsurface utilities, utility hazards associated potential unknown utilities that could be encountered (electrical, natural gas, etc.), and the physical and environmental hazards to have a spotter in proximity to the excavation. Spotters, if used, should be briefed on the potential physical and utility hazards that may be present at the site and the signs of subsurface utilities that they should be monitoring for during ground disturbance activities.

Uniform color codes for marking of underground facilities are provided in Attachment C.

Attachment A

Subsurface Utility Clearance Checklist

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SUBSURFACE UTILITY CLEARANCE CHECKLIST

Site Identification: _____

Project Consultant/Contractor: _____

Section 1: Safety, Preparation Tasks, and Mark-Outs

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Health and Safety Plan is available and all contractors and subcontractors are familiar with it.				
All applicable local, state, and federal permits have been obtained.				
Site access/permission has been secured.				
Most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) obtained.				
Reviewed site information to identify subsurface structures relevant to planned site activities (easements, rights-of-way, historical plot plans, fire insurance plans, tank dip charts, previous site investigations, soil surveys, boring logs, aerial photographs, etc.).				
Utility mark-outs have been performed by public utility company(s). Mark-outs clear/visible.				
Subsurface structure mark-outs performed by private mark-out company. Mark-outs clear/visible.				
Additional Activities: Were dig locations reviewed with site representative?				

Section 2: Initial Site Visit and Selecting Ground Disturbance Locations

Activity	Yes	No	N/A	Comments, including Justification if Response Is No or Not Applicable
Location of all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area has been identified.				
Location of utility mark-outs by all utility companies previously contacted has been identified within required time period.				
Location of all subsurface structure mark-outs by private mark-out company has been identified within required time period.				
Location of area lights/signs and associated subsurface lines identified.				
Location of all phones and associated subsurface lines identified.				
Location of all drains and associated interconnecting lines identified.				
Location of all electrical junction boxes and associated interconnecting lines identified				
Location of all natural gas meters or connections and all interconnecting lines identified				

Completed by: _____
Name

Signature: _____ _____
Company Date

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Attachment B

811 Utility Locate Brochure

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ALWAYS CALL BEFORE YOU DIG

One free, easy call gets your utility lines marked AND helps protect you from injury and expense.

Know what's below. Always call 811 before you dig. Visit call811.com for more information.

811
Know what's **below.**
Call before you dig.

INSERT CALL CENTER LOGO HERE

U.S. Department of Transportation

JOHN DEERE

TRAVELERS

Common Ground Alliance

Q: WHAT IS 811?

A: 811 is a new federally-mandated N-11 number designated by the FCC to consolidate all local "Call Before You Dig" numbers and help save lives by minimizing damages to underground utilities. One easy phone call to 811 quickly and easily begins the process of getting underground utility lines marked. Local One Call Center personnel will then notify affected utility companies, who will continue to mark underground lines for free.

Know what's **below.**
Call before you dig.

Q: WHY SHOULD I CALL 811 BEFORE EVERY DIG?

A: Calling 811 will help save lives and protect infrastructure. Knowing where underground utility lines are buried before each digging project begins helps protect you from injury, expense and penalties. The depth of utility lines varies and there may be multiple utility lines in the same area. Even simple digging projects can damage utility lines and can disrupt vital services to an entire neighborhood, harm diggers, and potentially result in expensive fines and repair costs. Marked lines show diggers the approximate location of underground lines and help prevent undesired consequences.

Q: I'M JUST A HOMEOWNER, NOT A CONTRACTOR—IS 811 FOR ME?

A: Calling 811 is for professional excavators and do-it-yourself homeowners. A recent national survey revealed that roughly half of Americans are "active diggers" who have done (or are planning to do) some type of digging project at home. Whether you are a professional excavator or an avid do-it-yourselfer, you need to call 811 before every dig every time.

Q: WHO IS PROMOTING AWARENESS OF 811?

A: The national 811 campaign is a project of The Common Ground Alliance (CGA), working with its 1,400 individual members, member organizations, sponsors and 811 campaign national launch partners. CGA is a member-driven association dedicated to ensuring public safety, environmental protection, and the integrity of services by promoting effective damage prevention practices. In recent years, the association has established itself as the leading organization in an effort to reduce damages to all underground facilities in North America through shared responsibility among all stakeholders.

Attachment C

Uniform Color Codes for Marking of Underground Facilities

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APWA UNIFORM COLOR CODE

WHITE - Proposed Excavation

PINK - Temporary Survey Markings

RED - Electric Power Lines, Cables, Conduit and Lighting Cables

YELLOW - Gas, Oil, Steam, Petroleum or Gaseous Materials

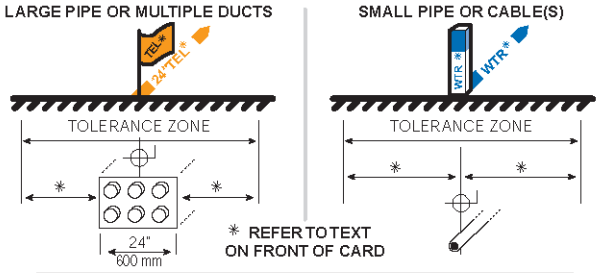
ORANGE - Communication, Alarm or Signal Lines, Cables or Conduit

BLUE - Potable Water

PURPLE - Reclaimed Water, Irrigation and Slurry Lines

GREEN - Sewers and Drain Lines

TYPICAL MARKING



Customize with your center's phone and address information

GUIDELINES FOR UNIFORM TEMPORARY MARKING OF UNDERGROUND FACILITIES

This marking guide provides for universal use and understanding of the temporary marking of subsurface facilities to prevent accidents and damage or service interruption by contractors, excavators, utility companies, municipalities or any others working on or near underground facilities.

ONE-CALL SYSTEMS
The One-Call damage prevention system shall be contacted prior to excavation.

PROPOSED EXCAVATION
Use white marks to show the location, route or boundary of proposed excavation. Surface marks on roadways do not exceed 1.5" by 18" (40 mm by 450 mm). The facility color and facility owner identity may be added to white flags or stakes.

USE OF TEMPORARY MARKING
Use color-coded surface marks (i.e., paint or chalk) to indicate the location or route of active and out-of-service buried lines. To increase visibility, color coded vertical markers (i.e., stakes or flags) should supplement surface marks. Marks and markers indicate the name, initials or logo of the company that owns or operates the line, and width of the facility if it is greater than 2" (50 mm). Marks placed by other than line owner/operator or its agent indicate the identity of the designating firm. Multiple lines in joint trench are marked in tandem. If the surface over the buried line is to be removed, supplementary offset markings are used. Offset markings are on a uniform alignment and clearly indicate the actual facility is a specific distance away.

TOLERANCE ZONE
Any excavation within the tolerance zone is performed with non-powered hand tools or non-invasive method until the marked facility is exposed. The width of the tolerance zone may be specified in law or code. If not, a tolerance zone including the width of the facility plus 18" (450 mm) measured horizontally from each side of the facility is recommended.

ADOPT UNIFORM COLOR CODE
The American Public Works Association encourages public agencies, utilities, contractors, other associations, manufacturers and all others involved in excavation to adopt the APWA Uniform Color Code, using ANSI standard Z535.1 Safety Colors for temporary marking and facility identification.

Rev. 4/99

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Standard Operating Procedure No. 004
for
Sample Packing and Shipping

Prepared by

EA Engineering, Science, and Technology, Inc., PBC
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 1
September 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 004	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	25 September 2018	Systematic update and review	Cristina Radu, Amanda Kohn	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of environmental samples to the laboratory for analysis. Additional requirements are applicable when shipping samples under the U.S. Environmental Protection Agency's Superfund Contract Laboratory Program.

NOTE: Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated could require shipment as dangerous goods; procedures for shipping of such samples are not covered in this SOP.

2. MATERIALS

The following materials may be required:

- Clear tape
- Custody seals
- Ice
- Packing material
- Plastic garbage bags
- Sample documentation
- Waterproof coolers (hard plastic)
- Zip-seal plastic bags.

3. PROCEDURE

Refer to SOP Numbers (Nos.) 001, 002, 016, and 039 as applicable.

Samples will be placed in clean, bubble-wrap lined sample coolers with double-bagged ice immediately after collection to ensure proper preservation. Most sample analyses require that the sample material is maintained at 2-6 degrees Celsius (°C). It is also important to ensure that sample containers are maintained at all times at the temperature required by the analytical method used to analyze the sample media; as such, samples should be retained in a chilled cooler during the inventory, quality control, and packaging process.

Check cap tightness and wipe down outside of each sample container. Verify that information on sample labels is correct and matches chain-of-custody forms. Ensure that both waterproof labels and indelible ink are used to label sample containers. Clear tape should be placed completely over the label. Wrap breakable sample containers in bubble wrap. Enclose each sample in a clear zip-seal plastic bag.

Prepare cooler for shipping. Empty any water that has accumulated in coolers from melting ice. Securely seal all valves and/or drain holes in the shipping container, both inside and out, with duct tape to prevent leakage in the event of sample container breakage or melting ice. Place several layers of bubble wrap on top of absorbent material and line the cooler sidewalls with bubble wrap. Line cooler with open garbage bag.

Prepare sample containers for shipping as follows:

- ***Glass Containers***—Wrap each glass sample container in bubble wrap or closed cell foam sheets. It is acceptable to package up to three 40-milliliter vials in one bubble wrap bag that is usually provided by the analytical laboratory. Enclose sample containers in a clear zip-seal plastic bag.

- ***Polyethylene Containers***—Place sample containers in clear zip-seal bags.

- ***Zip-Seal Bags***—Double-bag the samples to ensure that moisture will not reach the label.

Place all the sample containers upright inside garbage bag. Do not stack glass containers or lay them on their sides. Add additional bubble wrap between and around sample containers as needed to ensure containers do not shift during transport. If a second garbage bag was used, tie the (inner) garbage bag to isolate samples.

Double bag and seal loose, fresh ice to prevent melting ice from soaking the packing material. Fill gallon-size or larger zip-seal bags with fresh ice about two-thirds full and squeeze excess air out of the bags before sealing. Turn bag upside down and place in a second zip-seal bag, also removing excess air. Prepare sufficient bags to cover sample containers and ensure that the proper temperature (2-6° C) is maintained during transport.

Place ice on top of sample containers. Ensure that packing material does not insulate samples from ice. Do not use loose ice in sample coolers. Do not use bagged ice as packing material between or around sample bottles. Tie the garbage bag ensuring that the cooler lid will close securely.

Place a temperature blank into the cooler. The temperature blank consists of a plastic bottle containing either potable or deionized water. Temperature blanks are typically provided by the analytical laboratory. If temperature blanks are not provided, field staff must add a clean container filled with deionized water; ensure the cap is tight and container is labeled before placing in cooler.

If aqueous volatile organic analyte samples are being submitted, ensure a trip blank sample set is placed in each cooler containing volatile organic analyte samples. Trip blanks are used to check for contamination of volatile organic compound samples during handling, storage, and shipment from field to laboratory. The trip blanks consist of volatile organic analyte vials filled with deionized water and are typically provided by the analytical laboratory. Ensure that the trip blank samples and analyses are included on the chain-of-custody record.

Make copies of sample documentation (chain-of-custody forms or other field records) and retain in field files for record. Enclose the original field documentation forms in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

Seal coolers with signed and dated custody seals such that if the coolers were opened, the custody seals would be broken. Place clear tape over the custody seals to prevent damage to the seals.

Tape the cooler shut with packing tape over the hinges and custody seals. Tape should be wrapped around the cooler a minimum of five times. Ship all samples via overnight delivery on the same day they are collected if possible. Project-specific shipping requirements (e.g., Saturday delivery, communication with the receiving laboratory, etc.) should be discussed with the sample manager or project manager during project planning.

After samples are packaged within shipping containers, place shipping labels clearly on the outside of the container; clearly mark the number of containers in the shipment on the shipping label. Mark each cooler as "1 of 2," "2 of 2," etc.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

The project manager and field team leader are responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample meets or is suspected to meet the definition of "dangerous goods" per the Dangerous Goods Regulation of the International Air Transport Association, then that sample must be handled according to the instructions given for that material. Dangerous goods must be prepared for shipping only by personnel trained and certified by International Air Transport Association in dangerous goods shipment.

6. REFERENCES

Not applicable.

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Standard Operating Procedure No. 005
for
Field Decontamination

Prepared by

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Revision 2
September 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-*specific* variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 005	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	25 September 2018	Systematic update and review	Cristina Radu, Amanda Kohn	Matthew Bowman

1. SCOPE AND APPLICATION

All personnel or equipment involved in intrusive sampling, or that enter a hazardous waste site, must be thoroughly decontaminated prior to leaving the site to minimize the spread of contamination and prevent adverse health effects. This Standard Operating Procedure (SOP) describes the normal decontamination of sampling equipment and site personnel. Specific projects and programs may have additional decontamination requirements. Refer to the planning document(s) for additional site-specific requirements.

As a good practice, sampling at a site should be conducted moving from least to most impacted locations to minimize the potential for cross-contamination. It is advisable to use disposable tools and personal protective equipment to the extent possible such that decontamination is not necessary. If disposable equipment cannot be used, all attempts will be made to minimize the need for decontamination by using dedicated equipment when practical.

1.1 MATERIALS

The following materials may be required:

0.01 normal (N) hydrochloric acid	Non-phosphate laboratory detergent (Liquinox)
0.10 N nitric acid	Plastic garbage bags
Aluminum foil or clean plastic sheeting	Plastic sheeting, buckets, etc. to collect washwater and rinsates
Approved water (deionized, potable, etc.)	Pressure sprayer, spray bottles, brushes, laboratory wipes, disposable cloth (shop towel or similar)
High performance liquid chromatography (HPLC)-grade water[a]	Reagent grade alcohol[b]

a. For the purpose of this SOP, HPLC-grade water is considered equivalent to "deionized ultra-filtered water," "reagent-grade distilled water," and "deionized organic-free water." The end product is water that is pure with no spurious ions or organics to contaminate the sample. The method of generation is left to the individual contractor.
b. For the purpose of this SOP, the term "reagent grade alcohol" refers to either pesticide grade isopropanol or reagent grade methanol.

1.2 PROCEDURE

All reusable (non-dedicated) equipment that contacts or could potentially contact environmental samples shall be decontaminated prior to use at a site, between sampling locations, and at the completion of sampling events before leaving the site. Decontamination procedures are conducted in the Contaminant Reduction Zone, which may or may not be contiguous to the Exclusion Zone. The Contaminant Reduction Zone should be located on a level, preferably paved surface, either in an area upwind of the investigation/sampling area or in an area believed to be free of surface contamination. Care must be employed when moving contaminated tools and equipment to the Contaminant Reduction Zone to prevent the spread of contamination.

Specially designated and properly built decontamination pads may be built at a centralized location to accommodate larger pieces of equipment. The pads are built such that any water produced during the decontamination process can be contained and pumped into

investigative-derived waste holding containers (i.e., frac tank, 55-gallon drum, etc.) for waste profiling and disposal.

For other field equipment, the Contaminant Reduction Zone may be a mobile decontamination station set up in the vicinity of the Exclusion Zone or sampling location. Plastic sheeting will be used to create a clean surface for the sampling and decontamination equipment to be placed upon.

1.2.1 Sample Bottles

At the completion of each sampling activity, the exterior surfaces of the sample bottles must be decontaminated as follows:

- Ensure the bottle lids are on tight.
- Wipe the outside of the bottle with a paper towel to remove gross contamination.

1.2.2 Personnel Decontamination

Review the Health and Safety Plan for the appropriate decontamination of site personnel and reusable personal protective equipment, such as protective suits used at highly contaminated sites, respirators, safety boots, safety glasses, etc. Decontamination will be conducted in a designated Contaminant Reduction Zone as per the Health and Safety Plan and the general decontamination procedures outlined further in this SOP.

1.2.3 Non-Dedicated Equipment

Reasonable attempts will be made to minimize the need for decontamination by using dedicated equipment when practical.

All reusable (non-dedicated) equipment that contacts or could potentially contact environmental samples shall be decontaminated prior to use at a site, between sampling locations, and at the completion of sampling events before leaving the site. Decontamination shall be conducted at a central decontamination station (i.e., decontamination pad) or at the sampling location.

Decontamination stations should be located on a level, preferably paved surface, either in an area upwind of the investigation area or in an area believed to be free of surface contamination. Plastic sheeting will be used to create a clean surface for the sampling and decontamination equipment to be placed upon.

Used decontamination solutions will be disposed of properly according to the site-specific Health and Safety Plan or applicable planning documents.

1.2.3.1 Field Monitoring and Testing Equipment

Water quality meters and temperature, pH, conductivity, redox, and dissolved oxygen probes will be cleaned per the manufacturer's instructions. If no such specifications exist, remove gross contamination and triple rinse probe with HPLC-grade water. If downhole probes are used, wipe the wetted portion of the cable with a clean laboratory wipe or disposable cloth (shop towel or similar) that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination and rinse with approved water.

Electronic water level indicators, weighted tapes, measuring tapes transducers, level loggers, etc. will be decontaminated after each use as follows:

- Wipe the wetted or contaminated portion of the tape or cable and the probe with a clean laboratory wipe or disposable cloth (shop towel or similar) that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. Rinse cloth in the solution and continue wiping until tape or cable is clean.
- Wipe with a second wipe or cloth or rinse with HPLC-grade water to remove soap residue.
- Dry tape with a third cloth (or laboratory wipe) and rewind into case or on spool, or re-coil tape.

Other field monitoring or measuring equipment such as beakers and graduated cylinders used to measure flow rates; flow-through cells used for monitoring water quality parameters; piezometers used to determine water levels; packers, mechanical slug device, and downhole equipment used during aquifer (hydraulic) testing; etc. will be decontaminated by washing with a non-phosphate laboratory detergent solution, followed by approved water and HPLC-grade water rinse.

1.2.3.2 Bladder Pumps

Non-dedicated bladder pumps with disposable bladders will be decontaminated as follows:

- Disconnect tubing from pump.
- Completely disassemble the pump, being careful to note the initial position of and retain any springs and loose ball checks.
- Discard the pump bladder.
- Clean all parts in the same manner as provided in Section 1.2.3.1.
- Install a new Teflon® bladder and reassemble pump.

- Store pump in a clean, dedicated polyvinyl chloride, polytetrafluorethylene (PTFE), or low density polyethylene (for perfluorooctanesulfonic acid/per- and polyfluoroalkyl substances sampling) storage container.

1.2.3.3 Grundfos Redi-Flow® or Similar Submersible Pumps

Non-dedicated Grundfos Redi-Flow® and similar pumps will be disassembled and decontaminated per the manufacturer's instructions on an as-needed basis (i.e., where high concentrations and an elevated risk of cross-contamination exist). Due to the challenges associated with pump decontamination, if possible, consider designating one pump for sampling in highly contaminated areas and a second pump for sampling non-impacted areas or areas with lower contaminant concentrations. In most cases, the pumps will be decontaminated following the procedures below.

The pump and support cable/electrical wires that come in contact with water will be decontaminated via pumping as detailed below. To avoid electrical shock, always disconnect power from the pump when handling the pump body during decontamination procedures.

- Disconnect sample tubing from pump.
- Decontaminate the wetted portion of the cable/electrical wires by washing with non-phosphate laboratory detergent solution, followed by approved water and HPLC-grade water rinse. Coil cable/electrical wires on spools or clean plastic sheeting.
- Scrub the exterior of the pump to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent (steam cleaning may be substituted for detergent scrub).
- Transfer pump to rinse bucket filled with approved water. Rinse by pumping no less than nine volumes or a minimum of 5 minutes of approved water.
- Rinse pump exterior with reagent grade alcohol.
- Rinse pump exterior with HPLC-grade water.
- Rinse pump exterior with 0.10 N nitric acid solution
- Rinse pump exterior with HPLC-grade water.
- Allow pump to air dry.
- Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.
- Prior to reusing pump, rinse exterior again with HPLC-grade water.

1.2.3.4 Other Liquid Sampling Equipment

Other sampling equipment used to collect surface water, groundwater, non-aqueous phase liquid (NAPL), or other liquid samples includes but is not limited to PTFE double-check valve bailers, dip samplers (whether bucket, long-handled, or short-handled), discrete interval stainless-steel samplers, ball check valves and foot valves, and labware (i.e., beakers, graduated cylinders, vials, and other containers that are used to hold samples for field measurements/screening and water chemistry). This equipment will be decontaminated after each use as follows:

- Discard all ropes, tubing, etc. used in sampling in a properly marked sealable container, or as directed by the Health and Safety Plan. NOTE: No tubing is to be used in conjunction with a bailer in collecting samples.
- Wash sampling equipment with non-phosphate laboratory detergent and approved water solution using appropriate brush(es), laboratory wipes, or disposable cloth (shop towel or similar) to remove gross (visible) contamination.
- Rinse with approved water.
- Rinse with reagent grade alcohol.
- Rinse with HPLC-grade water.
- Rinse with 0.10 N nitric acid solution using a spray bottle. This rinse may be eliminated if inorganic compounds such as metals are not being sampled/are not a contaminant of concern.
- Rinse with HPLC-grade water.
- Allow equipment to air dry. If sampling equipment has just been used for purging and is being decontaminated prior to sampling, do not air dry. Double rinse with HPLC-grade water and proceed to collect samples.
- Wrap equipment in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.
- Rinse equipment with HPLC-grade water immediately prior to re-use.

1.2.3.5 Solid Materials Samplers

Solid materials samplers include soil and sediment sampling probes, augers, trowels, shovels, sludge samplers, and other sampling equipment (e.g., core tubes, grab samples, core catchers, core liners, scoops, spoons, etc.), which will be decontaminated as follows:

- Scrub the sampler to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate laboratory detergent (steam cleaning may be substituted for detergent scrub).
- Rinse off detergent with approved water.
- Rinse sampler with reagent grade alcohol.
- Rinse sampler with HPLC-grade water.
- For non-metallic samplers only, rinse sampler with 0.10 N nitric acid solution.
- For non-metallic samplers only, rinse sampler with HPLC-grade water.
- Allow sampler to air dry.
- Wrap sampler in aluminum foil or clean plastic sheeting, or store in a new zip-seal bag (size permitting) or clean, dedicated polyvinyl chloride or PTFE storage container.
- Rinse sampler with HPLC-grade water immediately prior to re-use.

For larger sediment sampling equipment, if sediment can be collected from the interior of a sampling device and away from potentially contaminated surfaces of the sampler, a site water rinse may be sufficient between stations. A site water rinse may also be sufficient for vessel surfaces between sample locations. However, all tools and equipment coming into contact with the sample should be decontaminated in accordance with the procedures above. Washwater from decontamination activities should be collected and disposed of properly.

1.2.3.6 Other Sampling and Measurement Probes

Soil (or sediment) gas sampling probes will be decontaminated as solids sampling devices.

1.2.3.7 Drilling Rigs, Sediment Sampling Vessels, and Other Heavy Equipment

All drilling rigs, sediment sampling vessels, and associated equipment such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out) will be decontaminated prior to site entry after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed prior to site entry when there is a likelihood that contamination has accumulated on tires and as spatter or dust on the way from one site to the next.

- Place contaminated equipment in an enclosure (i.e., existing wash pad, decontamination pad, etc.) designed to contain all decontamination residues (water, sludge, etc.).

- Steam clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) have been removed.

- Water used will be taken from an approved source.

- Containerize decontamination fluids in 55-gallon drums; sample; characterize; and, based on sample results, dispose of all decontamination residues properly.

Other heavy equipment includes use of backhoes, excavators, skid steers, etc. If heavy equipment is utilized during field activities (i.e., a backhoe for test pitting), the bucket should not come in contact with soil to be sampled. If the bucket contacts the soil to be sampled, then it should be decontaminated between sample locations, following the same procedures as listed above for a drill rig.

1.2.3.8 Ice Chests and Reusable Shipping Containers

Scrub exterior/interior with approved brush and Liquinox detergent. Rinse off detergent with approved water. Let air dry and properly store until re-use.

NOTE: If container/ice chest is severely contaminated, clean as thoroughly as possible, render unusable, and properly dispose of.

2. PRECAUTIONS

Segregate all waste streams as specified in the sampling documents and store investigation-derived waste properly. Dispose of all washwater, rinse water, rinsates, and other sampling wastes (tubing, plastic sheeting, etc.) in properly marked, sealable containers, or as directed by the Health and Safety Plan or applicable planning documents.

Once a piece of equipment has been decontaminated, be careful to keep it in such condition until needed.

3. REFERENCES

Site-specific Health and Safety Plan and/or applicable planning documents.

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Standard Operating Procedure No. 006
for
Summa Canister Sampling

Prepared by

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Revision 1
December 2014

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CONTENTS

Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to describe a procedure for the sampling of volatile organic compounds (VOCs) in ambient air. The method is based on samples collected as whole air samples in Summa-passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography and measured by mass-selective detector or multidetector techniques. This method presents procedures for sampling into canisters at final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling). This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. The organic compounds that have been successfully collected in pressurized canisters by this method are listed in the VOC Data Sheet below:

Compound (Synonym)	Formula	Molecular Weight	Boiling Point (°C)	Melting Point (°C)	CAS Number
Freon 12 (Dichlorodifluoromethane	Cl_2CF_2	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene	CH_2=CHCl	62.50	-13.4	-1,538.0	75-01-4
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
Dichlormethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	CH_3CHC1_2	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	CHCl=CHCl	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	-53.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2C1$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH_3CCl_3	`133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$ClCH=CCl_2$	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (*cis*-1,3-Dichloropropylene)	CH_3CCl=CHCl	110.97	76		
trans-1,3-Dichloropropene (*cis*-1,3-Dichloropropylene)	$ClCH_2CH$=CHCl	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	CH_2 $ClCHCl_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$Cl_2C=CCl_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C_6H_5Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3\text{-}(CH_3)_2C_6H_4$	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	$1,4\text{-}(CH_3)_2C_6H_4$	106.17	138.3	13.3	
Styrene (Vinyl benzene)	$C_6H_5CH=CH_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2\text{-}(CH_3)_2C_6H_4$	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5\text{-}(CH_3)_3C_6H_6$	120.20	164.7	-44.7	108-67-8

Compound (Synonym)	Formula	Molecular Weight	Boiling Point (°C)	Melting Point (°C)	CAS Number
1,2,4-Trimethylbenzene (Pseudocumene)	$1,2,4\text{-}(CH_3)_3C_6H_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3\text{-}Cl_2C_6H_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride (*a*-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2\text{-}Cl_2C_6H_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4\text{-}Cl_2C_6H_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	$1,2,4\text{-}Cl_3C_6H_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)					

These compounds have been measured at the parts per billion by volume level. These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations, or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency endorsement or recommendation for use.

2. METHOD SUMMARY

Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. Both modes may also use a mass flow controller/vacuum pump arrangement to regulate flow. With the above configuration, a sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated Summa-passivated canister. Alternatively, subatmospheric pressure sampling may be performed using a fixed orifice, capillary, or adjustable micrometering valve in lieu of the mass flow controller/vacuum pump arrangement for taking grab samples or short duration time-integrated samples. Usually, the alternative types of flow controllers are appropriate only in situations where screening samples are taken to assess for future sampling activities.

3. SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to a laboratory for analysis. Upon receipt at the laboratory, the canister tag data are recorded. Sample holding times and expiration should be determined prior to initiating field activities.

4. INTERFERENCES AND POTENTIAL PROBLEMS

Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned.

5. EQUIPMENT/APPARATUS

The following equipment/apparatus (Appendix A) is required.

5.1 SUBATMOSPHERIC PRESSURE SAMPLING EQUIPMENT

1. ***VOC Canister Sampler***—Whole air sampler capable of filling an initially evacuated canister by action of the flow controlled pump from vacuum to near atmospheric pressure (Andersen Samplers Inc., Model 87-100 or equivalent).

2. ***Sampling Inlet Line***—Stainless steel tubing to connect the sampler to the sample inlet.

3. ***Sample Canister***—Leak-free stainless steel pressure vessels of desired volume with valve and Summa-passivated interior surfaces (Scientific Instrumentation Specialist, Inc., ID 83843, Andersen Samplers, Inc., or equivalent).

4. ***Particulate Matter Filter***—2-micrometer (µm) sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).

5. ***Chromatographic Grade Stainless Steel Tubing and Fittings***—For interconnections (Alltech Associates, Cat. No. 8125, or equivalent). All materials in contact with sample, analyte, and support gases should be chromatographic grade stainless steel.

6. ***Fixed Orifice, Capillary, or Adjustable Micrometering Valve***—Used in lieu of the electronic flow controller/vacuum pump for grab samples or short duration time-integrated samples.

5.2 PRESSURIZED SAMPLING EQUIPMENT

1. ***VOC Canister Sampler***—Whole air sampler capable of filling an initially evacuated canister by action of the flow controlled pump from vacuum to near atmospheric pressure (Andersen Samplers Inc., Model 87-100).

2. ***Sampling Inlet Line***—Stainless steel tubing to connect the sampler to the sample inlet.

3. ***Sample Canister***—Leak-free stainless steel pressure vessels of desired volume with valve and Summa passivated interior surfaces (Scientific Instrumentation Specialist, Inc., ID 83843, Andersen Samplers, Inc., or equivalent).

4. ***Particulate Matter Filter***—2-µm sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).

5. ***Chromatographic Grade Stainless Steel Tubing and Fittings***—For interconnections (Alltech Associates, Cat. No. 8125, or equivalent). All materials in contact with sample, analyte, and support gases should be chromatographic grade stainless steel.

6. REAGENTS

Not applicable.

7. PROCEDURE

7.1 SUBATMOSPHERIC PRESSURE SAMPLING

7.1.1 Sampling Using a Fixed Orifice, Capillary, or Adjustable Micrometering Valve

1. Prior to sample collection, the appropriate information is completed on the Summa Air Sampling Work Sheet (Appendix B).

2. A canister, which is evacuated to 0.05-millimeter Hg and fitted with a flow restricting device, is opened to the atmosphere containing the VOCs to be sampled.

3. The pressure differential causes the sample to flow into the canister.

4. This technique may be used to collect grab samples (duration of 10-30 seconds) or time-integrated samples (duration of 12-24 hours). The sampling duration depends on the degree to which the flow is restricted.

5. A critical orifice flow restrictor will have a decrease in the flow rate as the pressure approaches atmospheric.

6. Upon sample completion at the location, the appropriate information is recorded on the Summa Air Sampling Work Sheet. VOCs are to be sampled.

7.1.1.1 Sampling Using a Fixed Orifice Valve Summa Canister

1. Before leaving to the field and receiving the sample canister, field personnel will need to verify in the Sampling and Analysis Plan the type of sample or samples to be collected.

2. Before leaving to the field and receiving the sample canister, field personnel will need to verify in the Sampling and Analysis Plan the total amount of time the sample canister will need to be deployed at the sample collection location to collect the actual air sample.

3. Before leaving to the field and receiving the sample canister, field personnel will need to contact and verify with the laboratory providing the sample canister that the flow controller and critical orifice provided in the sample canister will allow for the type of sample and the total amount of time needed to collect the sample requested in the Sampling and Analysis Plan.

   Field personnel will also need to ask the laboratory providing the sample canister if the sample canister has a built-in vacuum gauge and if the flow controller has a built-in vacuum gauge. The importance of a vacuum gauge is identified in Steps 5 and 7.

4. Upon receiving the sample canister, field personnel will need to inspect the sample canister shipment and familiarize themselves with the sample canister and equipment associated with the sample canister.

   Typical items associated with a sample canister are: pre-filter, flow controller, flow controller vacuum gauge, sample canister valve, sample canister inlet, sample canister vacuum gauge, brass dust cap, and rain guard.

5. Before sampling is started and equipment is attached to the sample canister, record the sample canister vacuum gauge reading, the canister's serial number, and the flow controller serial number on the appropriate field data form.

   NOTE: There are some instances when a sample canister is not equipped with a built-in sample canister vacuum gauge (Step 7).

6. Attach a string tied sample container tag to the frame of the sample canister. At a minimum, the sample container tag should contain the: sample name, analysis method, sample date, sampler, and sample time.

   NOTE: Do not attach adhesive-type labels to the sample canister.

7. When ready to sample, remove the brass dust cap from the sample canister inlet.

   If a test gauge is to be used to record the sample canister vacuum, attach the gauge to the sample canister inlet and record the sample canister vacuum reading on the appropriate field data form. See Steps 8, 9, and 10 for equipment tightening information.

   In the event a sample canister is not equipped with a built-in vacuum gauge, the laboratory will often provide a flow controller with a built-in vacuum gauge which can be used to record the sample canister vacuum.

8. Connecting equipment/flow controller to the sample canister inlet, tighten the equipment/flow controller fitting nut to the sample canister inlet finger tight, being careful not to cross the threads.

To ensure that the equipment/flow controller is being correctly installed onto the sample canister inlet, hold the flow controller and gently rotate the flow controller back and forth while finger tightening the flow controller fitting nut to the sample canister inlet.

9. Once the equipment/flow controller fitting nut is finger tightened to the sample canister inlet and it has been verified that the equipment/flow controller fitting nut threads are aligned properly to the sample canister inlet, using a flat profile wrench (typically a 9/16- or ½-in.), tighten the equipment/flow controller fitting nut.

   DO NOT use adjustable end wrenches or pliers to tighten a fitting nut.

   NOTE: To tighten an equipment/fitting nut, a 1/8 in. should be sufficient. DO NOT over-tighten any equipment/fitting connections. Over-tightening will cause leaks.

   IMPORTANT: DO NOT use Teflon™ tape or other sealants on any equipment/fitting threads; they are not necessary.

10. To begin sampling, open the sample canister valve by turning it counter-clockwise one full turn.

    NOTE: Canister valves can be of two types: rotary and toggle.

    Record the start time and the initial sample canister vacuum gauge reading on the appropriate field data form.

11. Field personnel need to periodically check the sample canister vacuum gauge throughout the sampling period to ensure that sufficient vacuum remains in the sample canister for the time the sample canister will need to be deployed to collect the sample.

12. Once the sampling period is complete, close the sample canister valve finger tight or snug. DO NOT over-tighten the sample canister valve as this may possibly damage the sample canister valve.

    NOTE: It is preferable to stop the sampling period when the sample canister vacuum gauge reads -5" Hg. Allowing the sampling period to extend past -5" Hg may possibly result in an unusable sample.

13. Record the stop time and the ending sample canister vacuum gauge reading on the appropriate field data form.

14. Disassemble the equipment/flow controller in reverse order of the above assembly instructions. Return all equipment/flow controller to the original packaging material and shipping container in the manner in which they were received.

15. Complete the chain-of-custody form, ensuring all fields are entered, repackage the sample canister and its associated equipment in the original shipping container, secure the outside of the shipping container with custody seals to ensure the integrity of the sample canister, and return the sample canister to the procured laboratory.

7.1.2 Sampling Using a Mass Flow Controller/Vacuum Pump Arrangement (Andersen Sampler Model 87-100)

1. Prior to sample collection, the appropriate information is completed on the Summa Air Sampling Work Sheet (Appendix B).

2. A canister is connected in line with the sampler and is opened to the atmosphere containing the VOCs to be sampled.
3. A whole air sample is drawn into the system through a stainless steel inlet tube by a direct drive blower motor assembly.

4. A small portion of this whole air sample is pulled from the inlet tube by a specially modified inert vacuum pump in conjunction with a mass flow controller.

5. The initially evacuated canister is filled by action of the flow controlled pump to near atmospheric pressure.

6. A digital time-program is used to pre-select sample duration and start and stop times.

7. Upon sample completion at the location, the appropriate information is recorded on the Canister Sampling Field Data Sheet.

7.2 PRESSURIZED SAMPLING

7.2.1 Sampling Using a Mass Flow Controller/Vacuum Pump Arrangement (Anderson Sampler Model 87-100)

1. Prior to sample commencement at the location, the appropriate information is completed on the Canister Sampling Field Data Sheet.

2. A canister is connected in line with the sampler and is opened to the atmosphere.

3. A whole air sample is drawn into the system through a stainless steel inlet tube by a direct drive blower motor assembly.

4. A small portion of this whole air sample is pulled from the inlet tube by a specially modified inert vacuum pump in conjunction with a mass flow controller.

5. The initially evacuated canister is filled by action of the flow controlled pump to a positive pressure not to exceed 25 pounds per square inch gauge.

6. A digital time-programmer is used to pre-select sample duration and start and stop times.

7. Upon sample completion at the location, the appropriate information is recorded on the Canister Sampling Field Data Sheet.

8. CALCULATIONS

A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined for the canister and is filled to approximately 88.1 kPa for subatmospheric pressure sampling or to approximately one atmosphere above ambient pressure for pressurized sampling over the desired sample period. The flow rate can be calculated by:

$$F = \frac{(P)(V)}{(T)(60)}$$

where

F = Flow rate (cm^3/min)
P = Final canister pressure, atmospheres absolute
V = Volume of the canister (cm^3)
T = Sample period (hours).

For example, if a 6-liter canister is to be filled to 202-kPa (two atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{(2)(6000)}{(24)(60)} * 8.3 cm^3/min$$

If the canister pressure is increased, a dilution factor is calculated and recorded on the sampling data sheet.

where

$$\text{Dilution Factor} = \frac{Ya}{Xa}$$

Xa = Canister pressure (kPa, pounds per square inch absolute) absolute before dilution.
Ya = Canister pressure (kPa, pounds per square inch absolute) absolute after dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

9. QUALITY ASSURANCE/QUALITY CONTROL

The following general quality assurance procedures apply (VIP-8, U.S. Environmental Protection Agency 600/9-87-010):

1. Data must be documented on standard volume sets when sampling ambient air: chain-of-custody records, field data sheets, or using solid adsorbents (atmospheric site logbooks. Environ. 18:855-859, 1984).

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the Work Plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and must be documented.

3. Duplicates, replicates, or other quality assurance/quality control samples may be collected as appropriate or as defined in project-specific Work Plans and Quality Assurance Project Plans.

10. DATA VALIDATION

Not applicable.

11. SAFETY AND HEALTH

When working with potentially hazardous materials, follow U.S. Environmental Protection Agency, Occupational Safety and Health Administration, and Corporate safety and health practices. Specifically, pressurization of Summa canisters should be performed in a well ventilated room, or preferably under a fume hood. Care must be taken not to exceed 40 pounds per square inch in the canisters. Canisters are under pressure, albeit only 20-30 pounds per square inch, and should not be dented or punctured. They should be stored in a cool dry place and always be placed in their plastic shipping boxes during transport and storage.

12. REFERENCES

McClenny, W.A., J.D. Pleil, T.A. Lumpkin, and K.D. Oliver. 1987. Update on Canister-Based Samplers for VOCs. Proceedings of the 1987 EPA/APCA Symposium on Measurement of Toxic and Related Air Pollutants. May. APCA Publication.

Riggin, R.M. 1983. Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air. EPA-600/4-83-027. U.S. Environmental Protection Agency, Research. Triangle Park, North Carolina.

Walling, J.F. 1986. The Utility of Distributed Air Monitoring VOC Sources. EPA-340/1-88-015, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Washington, D.C. June.

Appendix A

Equipment/Apparatus

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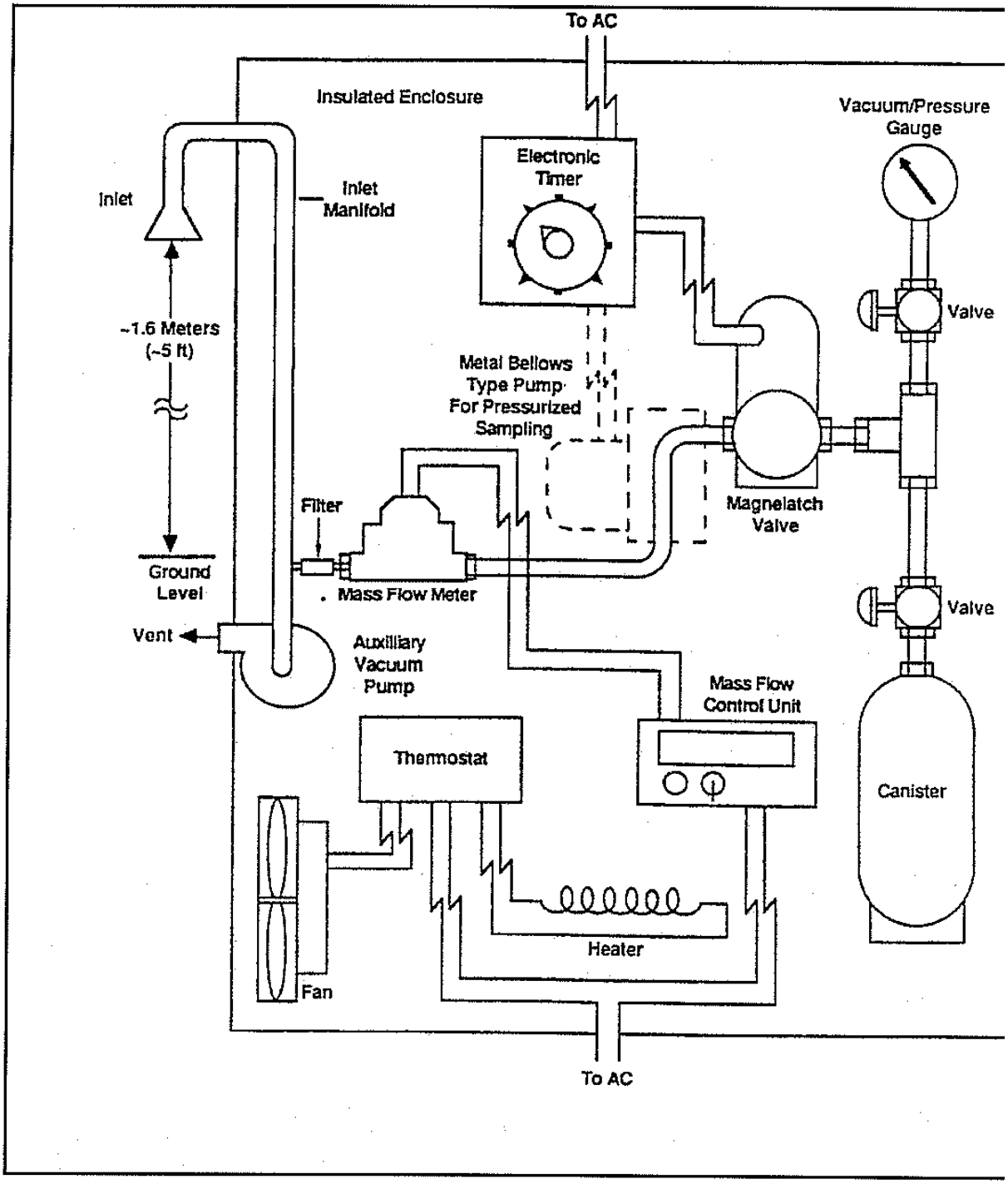
Appendix A

Subatmospheric/Pressurized Sampling Equipment



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Appendix B

Summa Air Sampling
Work Sheet

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APPENDIX B

SUMMA AIR SAMPLING WORKSHEET

Site:		**Site No.:**	
Samplers:		**Work Assignment Manager:**	
Date:		**Project Leader:**	

Sample No.					
Location:					
Summa ID					
Orifice Used					
Analysis/Method					
Time (Start)					
Time (Stop)					
Total Time					
SUMMA WENT TO AMBIENT	**Yes/No**	**Yes/No**	**Yes/No**	**Yes/No**	**Yes/No**
Pressure Gauge					
Pressure Gauge					
Flow Rate (Pre)					
Flow Rate (Post)					
Flow Rate (Avg.)					
MET Station Onsite: Yes/No					

General Comments:

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Standard Operating Procedure No. 010
for
Water Level and Well Depth Measurements

Prepared by

EA Engineering, Science, and Technology, Inc., PBC
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Revision 1
September 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 010	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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CONTENTS

DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	25 September 2018	Systematic update and review	Cristina Radu, Amanda Kohn	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to present the protocols for measuring depth to groundwater, presence and thickness of non-aqueous phase liquid (NAPL), and well depth in groundwater wells. This procedure is applicable to the sampling of monitoring wells and must be performed prior to any activities that may disturb the water level (i.e., purging or aquifer testing).

2. MATERIALS

The following materials may be required:

- Electronic sounding device with calibrated cable or tape measured at 0.01-foot increments (i.e., water level meter or oil/water interface probe) OR transducer and datalogger
- Plastic sheeting
- Photoionization detector or intrinsically safe flame ionization detector
- Materials required for decontamination per SOP Number (No.) 005
- Well construction diagrams, well records, and/or survey information
- Field forms (i.e., well gauging forms, well assessment forms, purge logs) and/or field logbook.

3. PROCEDURE

3.1 PRELIMINARY STEPS

Compile well construction data/forms, survey information, and historical data, if available, prior to field mobilization. The type and length of electronic sounding device to be used will be based on the monitoring well diameter, well installation depth, and the presence/absence of NAPL. Oil/water interface probes will be used in all wells for the first round of sampling, regardless of site history.

When planning on measuring depth to water at a site where product may be present in wells, the wells should be segregated between potentially contaminated and not contaminated categories. The sequence of well gauging should be established to minimize the potential of cross contamination by generally gauging clean wells first.

Ensure that the electronic sounding device is working prior to mobilization by submerging the probe in a container of potable or deionized water. Keep the indicator probe in its protective case when not in use. Locate the well and verify its position on the site map. Record on the applicable field form(s) or in the field logbook whether positive identification was obtained, including the well number/identification and any identifying marks, codes, or tags contained on the well casing or protective casing.

3.2 WELL HEADSPACE SCREENING

Refer to the Health and Safety Plan or applicable planning documents to determine if well headspace screening is required. At a site where historical information is available, well headspace screening may be omitted.

Headspace screening will be conducted using an organic vapor meter (photoionization detector or flame ionization detector). All headspace screening should be performed at arm's length and from the upwind side of the well if possible. Refer to SOP Nos. 011, 023, or 024 as appropriate.

Screen the ambient air in the breathing area around the wellhead and record the reading on the applicable field form(s) and/or in the field logbook. Once the breathing area is deemed safe, unlock/open the well protective casing to get access to the wellhead. Re-screen the ambient air again to determine if organic vapors may have accumulated.

Screen the air in the wellhead (headspace) for gross organic vapors. This will indicate the presence of gross volatile contaminants as well as potential sampler exposure. Most well casings are covered with a cap, some are outfitted with pump assemblies, while some may not have a cap at all. If a cap is present, sample the air in the wellhead for gross organic vapors by lifting the well cap only high enough for the organic vapor meter probe to be entered into the well casing. If a pump assembly is present on top of the casing, locate the gauging port, remove the cap, and insert the probe to make the measurement. If a cap is not present, insert the probe in the well casing. Record the reading on the applicable field form(s) and/or in the field logbook.

If volatiles are detected, allow the well to vent for 60-90 seconds and re-screen and record the headspace readings. If the second reading is lower than the first, use the second reading to determine whether respiratory protection will be required during subsequent activities.

3.3 WELL ASSESSMENT

Once the breathing zone at the wellhead is deemed safe or applicable respiratory protection is donned if needed, conduct the well assessment. Record the well assessment information on applicable field forms (well assessment form) or in the field logbook.

Assess and record the condition of the well casing, well pad and bollards, well cover, and any equipment (pump assembly). Record any observations and remarks regarding the completion characteristics and well condition (i.e., evidence of cracked casing or surface seals, security of the well [locked cap], or evidence of tampering). Note if there are discrepancies between current

well condition/completion and well construction diagrams/records or well survey data (i.e., damage or modifications to the well including but not limited to frost heaving, broken or otherwise damaged casing, conversion to/from flush mount or stick-up, installation or removal of polyvinyl chloride collar or other material on inner casing, installation or removal of a pump assembly, etc.).

Next, locate the measurement reference point from where water, NAPL, and well depth measurements will be performed. This reference point should be scribed, notched, or otherwise noted and the elevation will be recorded in the well survey data. It is critical that the actual survey point is known and used consistently throughout monitoring events.

If no reference marks are present or if changes have been made to the well casing since the survey, measure depths based on highest point of the well casing. If there is no high point, measure depths to the northern side of either the well polyvinyl chloride casing or the pump assembly cover. Permanently mark the measurement location for future survey and/or measurement purposes. Determine the new reference point elevation by measuring the distance from a known surveyed point (surveyed elevation of the protective casing or ground surface). Record this difference on the applicable field form(s) or in the field logbook for use in groundwater elevation calculations.

3.4 LIQUID LEVEL AND WELL DEPTH MEASUREMENTS

Typically, a complete round of static liquid levels and monitoring well depths is conducted as one of the first steps during groundwater monitoring. However, if monitoring wells are to be sampled for polyfluorinated alkyl substances, gauging should be completed after groundwater sampling to mitigate the possibility of cross-contamination.

Equipment should be decontaminated prior to first use in the field and after each use. Refer to SOP No. 005 for decontamination procedures. Keep all equipment and supplies protected from gross contamination; use clean plastic sheeting and keep the electronic sounding device probe in its protective case when not in use.

Measure NAPL and water levels and well depths as detailed in the subsections below. When measuring depths, grasp the cable with the thumb and forefingers at the top of the casing and record the depth based on the measurement reference point detailed in Section 3.3.

Gauging information including dates/times, water depths, NAPL depths and thicknesses, and well depths will be recorded on applicable field forms (i.e., well gauging form, well assessment form, purge form, etc.) and/or in the field logbook.

3.4.1 Non-Aqueous Phase Liquid Level Measurements

Always perform NAPL checks for the following conditions:

- The first time a well is sampled
- In wells installed in or near areas with suspected or confirmed NAPL contamination
- If headspace test reveals presence of volatiles.

Use an oil/water interface probe to determine the presence and thickness of NAPL. An oil/water interface probe will have a different alarm tone (continuous or intermittent) for NAPL versus water. The air/liquid interface depth measurements will be more accurate if the probe is lowered into liquid. The NAPL/water depths will be more accurate if the probe is moved from water into NAPL. Always lower and raise the interface probe slowly to prevent undue mixing of media. Complete all measurements as follows:

- Upon removing the well cap as a part of headspace screening described in Section 3.2, ensure that enough time (a couple of minutes) has passed for the air pressure in the well to have equalized with atmospheric pressure.

- Turn the interface probe on and test the alarm and liquid indication light.

- Remove the indicator probe from the protective case. Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the alarm sounds and the liquid indication light comes on.

- If LNAPL is detected on top of the water column, record the depth of the initial level/first alarm (top of the product layer). Continue to slowly lower the probe until it passes into the water phase. Slowly retract the probe until the NAPL alarm sounds and record the product/water interface depth (base of the product layer). Calculate and record the LNAPL thickness.

- Continue to slowly lower the interface probe through the water column to check for the presence of dense non-aqueous phase liquid (DNAPL). If DNAPL is encountered, measure and record the product interface depths (top and base of the DNAPL layer[s]) and calculate and record the DNAPL thickness(es).

- Continue lowering the probe until the base of the well is encountered. Measure the depth of the well as detailed in Section 3.4.3.

- Slowly raise the interface probe, recording the depth to each interface as the probe is withdrawn. While raising the probe, wipe the wetted portion of the tape with a clean laboratory wipe or disposable cloth (shop towel or similar) that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. If there is a discrepancy in depths, clean the probe sensors and re-measure the depths.

- Decontaminate the measuring tape and probe between well locations as detailed in SOP No. 005 to minimize the potential of cross contamination.

3.4.2 Water Level Measurements

If a well has been sampled previously and no NAPLs were present, or if none of the preceding NAPL check conditions are met, the NAPL check may be omitted and an electronic water level detector can be used to measure water levels.

- Upon removing the well cap, ensure that enough time (a couple of minutes) has passed for the air pressure in the well to have equalized with atmospheric pressure.

- Turn the water level meter and test the alarm and liquid indication light. Adjust the sensitivity scale as needed.

- Remove the water level indicator probe from the case, and slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the alarm sounds and the liquid indication light comes on. Very slowly, raise and lower the probe until the point is reached where the alarm just sounds. Record the depth to water.

- Slowly raise the probe and wipe the wetted portion of the tape (if any) with a clean laboratory wipe or disposable cloth (shop towel or similar) that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination.

- Decontaminate the measuring tape and probe between well locations as detailed in SOP No. 005 to minimize the potential of cross contamination.

3.4.3 Well Depth Measurements

The depth of a well is a stable value established during well construction; changes in well depth are usually indicative of a potential problem with the well. Fluctuations in well depth may be caused by either settlement of fine-grained material (i.e., silt) at the bottom of the well or damage to the well casing or screen.

Do not attempt to measure the depth of a well when a dedicated pump is installed in the casing. The weighted tape or the electric water level indicator will likely get snagged onto the tubing and damage the pump assembly. The depth of the well should also not be measured in wells in which passive diffusion samplers have been deployed; tag the bottom of the well after the samplers have been removed and before their re-deployment for the next sampling round.

A weighted tape is the preferred tool for measuring well depths. For shallow wells, an electronic water level indicator probe may be employed. In deeper wells, a weight may be attached to the probe to aid in measuring the well depth. Well depths will be measured as follows:

- Lower the probe until it is resting on the bottom of the well. Slowly pull upward on the tape until a tug can be felt while lifting the probe off the well bottom.

- Record the depth of the well. If the tape distance markings on the electronic sounding device are not marked to the end of the probe (i.e., markings are referenced to an electrode in the middle of the probe), add the length of the probe beneath the electrode to the measured depth to obtain the true depth of the well.

- Compare the recorded depth to the installation depth in the well construction diagram/record and note any discrepancies. If discrepancies exist, re-measure the well depth. Note the presence of sediment at the base of the well (i.e., hard bottom versus soft bottom).

- Withdraw the probe and tape. While raising the probe, wipe the wetted portion of the tape with a clean laboratory wipe or disposable cloth (shop towel or similar) that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination.

- Decontaminate the measuring tape and probe between well locations as detailed in SOP No. 005 to minimize the potential of cross contamination.

3.5 TRANSDUCERS AND DATALOGGERS

Transducers and dataloggers may be used for depth to water measurements in wells where water level fluctuations over time are to be measured, such as tidal fluctuation studies (SOP No. 045) and aquifer (hydraulic) tests (SOP No. 033). Note that transducers are inappropriate for measuring well depth.

No calibration is necessary before use. Depending upon the device used, correction factors may be required for some measurements. Check instrument batteries prior to each use. Exercise care not to break the seals at the top of the electric water level indicator probe.

3.5.1 Transducers Deployment

Attach the transducer umbilical leads to the datalogger. Turn datalogger on. Program the transducer following instructions provided in the instrument user manual. Refer to the planning document(s) for site-specific parameters and recording frequency.

Measure and record the depth to water and well depth using an electronic sounding device as detailed in Sections 3.4.2 and 3.4.3. Slowly lower the transducer into the well until it is below the lowest possible piezometric level (typically 2-3 feet below the water table). Attach the cable grip to the well protective casing and/or tape the cable to the casing to prevent the transducer from falling further.

Record the following information and computations in the field logbook during transducer deployment:

- Date and time of deployment
- Weather
- Casing elevation
- NAPL surface elevation = casing elevation – depth to NAPL
- NAPL thickness = depth to bottom of NAPL – depth to top of NAPL
- Water level elevation = casing elevation – depth to water
- Well bottom elevation = casing elevation – depth to bottom (or read directly from tape).
- Method of measurement.

With the transducer deployed and the umbilical secured to the protective casing, ensure that the transducer unit is programmed to start logging at a desired date and time, or manually start logging. Record the logging start time. View real-time readings using the data logger and download a series of data using the data logger to verify proper operation. If the transducer is logging as desired, allow the transducer to continue logging. If the data are not logging as required in the planning documents, stop data collection, re-program the transducer, and restart logging.

3.5.2 Transducer Data Recording and Manipulation

Periodically check and download data per the manufacturer's instructions at the frequency detailed in the planning document(s) using a datalogger or computer and instrument software to download the transducer. If data are downloaded onto a datalogger, upload the data to a computer upon returning to the office.

Use the transducer manufacturer's software and transducer deployment information to make the following updates to the transducer data as needed:

- Correct the raw water pressure data files from the submersible transducer(s) for barometric effects

- Convert the transducer-reported values to equivalent feet of water over the sensor

- Normalize the transducer water levels as depths to groundwater in feet below the water level measuring point.

3.5.3 Transducer Retrieval

Upon completion of data collection, withdraw the transducer and cable from the well. Decontaminate the transducer and cable as detailed in SOP No. 005.

4. SPECIAL CONSIDERATIONS

Measurement of depth to water in new wells should only be performed after the water elevation in the well has stabilized. This may take as long as 72 hours; however, if the formation in which the well was installed is tight, the well may take even longer to achieve steady state. Ensure that steady-state conditions have been reached before making measurements as determined by the project geologist.

Electronic sounding devices may sometimes give erroneous readings due to water droplets along the side of polyvinyl chloride casing or on sample/pump tubing within a well. To check for erroneous readings, raise the probe above the point where the first sound was noted; a continued buzzer alarm indicates that the water table has not been reached. Shake the tape to remove water adhered to the tape and continue lowering to the water table.

5. CALIBRATION

No calibration is required. However, the marked tapes of the instruments described in this SOP may stretch, especially when depth of the wells is great. If more than one instrument is used at a site during the same gauging event, consider comparing the markings of the tapes on all instruments by stretching them on clean plastic sheeting to the anticipated length to be used. If the delta is known between the tapes, corrections of the measurements can be done at the time data are processed.

6. PRECAUTIONS

Depending upon the device used, correction factors may be required for some measurements. Check instrument batteries prior to each use. Exercise care not to break the seals at the top of the electric water level indicator probe.

7. REFERENCES

Not applicable.

Standard Operating Procedure No. 013
for
Collection of Monitoring Well Samples

Prepared by

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 013	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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CONTENTS

DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	1/23/2019	Systematic update and review	Jason Stroup, Scott Dobson	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the collection of groundwater samples from monitoring wells.

2. MATERIALS

The following materials may be required:

0.45-µM filters	Polyvinyl chloride bailer (for purging only)
Bladder pump (dedicated to one well only)	Sample bottles and labels
Conductivity meter	Stainless steel bailer (for purging and sampling)
Dissolved oxygen meter	Submersible pump and hose (for purging only)
Generator	Thermometer (optional)[1]
Logbook or book of field parameter forms	Transparent bailer with a double check valve
Peristaltic pump with tubing for filtering samples	Turbidity meter
pH meter with oxidation-reduction potential probe	Poly or Teflon® and/or Teflon® lined tubing **(PTFE or Teflon® should not be used when sampling for PFCs/PFAS)**
Photoionization detector organic vapor analyzer.	Variable speed, low flow submersible pump (e.g., Grundfos MP1 groundwater sampling pump) (for purging and sampling)
Variable speed peristaltic pump	Peristaltic head tubing
Plastic sheeting	Water level indicator
Polypropylene rope	Interface probe

NOTES: µM = Micrometer(s).
L = Liter.
mL = Millimeter.
PFAS = Per- and polyfluoroalkyl substances.
PFC = Perfluorinated compound.
PTFE = Polytetrafluoroethelyne.
VOC = Volatile organic compound.
PTFE bailer with PTFE-coated stainless steel cable, double check valve top, and controlled flow bottom discharge attachment[2] for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles) (for purging and sampling)

3. PROCEDURE

3.1 GENERAL

Groundwater sampling will follow these general steps:

1. Temperature compensation and measurement capabilities are generally available as integral functions of pH meters and conductivity meters. If this is the case, a separate thermometer is not required.
2. Although use of a controlled flow bottom discharge valve is historically preferred, use of such a device can cause aeration of the sample.

- Arrive onsite
- Set up apparatus (generators, pumps, etc.)
- Glove
- Organic vapor check, water level, and well depth measurements
- Sample non-aqueous phase liquids (NAPLs) (as required)
- Begin purge procedure
  - If using bailer to purge and sample, see Section 3.6
  - If using pump to purge and bailer to sample, see Section 3.7
  - If using bladder or low-flow pump to purge and sample, see Section 3.8
- Decontaminate/re-glove
- Take samples
  - If with bailer, see Section 3.6
  - If with bladder or low flow pumps, see Section 3.8
- Decontaminate/dispose of wastes, move equipment to next site.

NOTE: Sampling monitoring wells that contain PFAS have specific requirements that must be followed. Review SOP No. 073 prior to planning or conducting any sampling for PFAS.

3.2 GENERAL RULES FOR GROUNDWATER FIELD PARAMETER LOGBOOK

Use only one site or installation per logbook, and only one sampling location per page or form (if using pre-printed forms). The same logbook may be used for more than one sampling event. The first five pages will be reserved for index, general notes, etc. Sign and date each entry. The last five pages will be reserved for recording calibration data for the pH, temperature, turbidity, oxidation-reduction potential, dissolved oxygen, and conductivity meters. Use the page number or a separately recorded "Cal Reference Number" to refer to each calibration.

As appropriate, insert the cardboard flap under the form being filled out so that writing does not go through to the pages below. As appropriate, fill in the forms from front to back of the logbook, tearing out the white copy for each sample when the sample has been collected. This copy goes in the cooler with the sample, directly to the laboratory. The original copy must be torn out before you write on the back of the duplicate form. As appropriate, duplicate copies, index pages, and calibration sheets remain intact.

Reference SOP No. 016 for additional procedures and requirements for the use and maintenance of field logbooks for sampling.

3.3 GROUNDWATER SAMPLING GENERAL RULES

Groundwater samples will be collected from the least contaminated wells first, progressing to the most contaminated[3]. Upon arrival at the well site, immediately set up and organize the purging, sampling, and filtration equipment. If needed, due to muddy or contaminated ground, remoteness from sampling vehicle, and/or for placement of hose(s) and/or power cord if a pump is used, place clean plastic sheeting at, or around the well, to serve as a clean staging area for purging and sampling equipment, as conditions warrant. Care must be exercised not to step on plastic sheeting. If the well is remote from the sampling vehicle, set up the filtration equipment and place rope, wrapped bailer, and pre-labeled sample containers on the plastic sheet from the well. When a pump is to be used, situate the portable generator on level ground approximately 15 feet (ft) away from and downwind from the well. All generator maintenance (oil and fueling) is to be performed offsite. If the hose(s) and/or power cord of the pump are not on a reel, place the pump with its hose and power cord on the plastic sheeting downhill from the well.

Check well headspace for organic vapor, which may pose a health and safety hazard and indicate the presence of NAPL. Measure depth(s) to and thickness(es) of NAPL(s) as appropriate. Measure the depth to water and depth of well. From the water depth, well diameter, sand pack length, etc., calculate the equivalent volume (1 EV) of water in the well.

$$1\ EV = \text{volume in casing} + \text{volume in saturated sand pack}$$

Therefore, if the water table lies below the top of the sandpack, use the following equation:

$$1\ EV = (\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_w) * (0.0043)$$

If the water table lies above the top of the sandpack use this equation:

$$1\ EV = [(\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_s)] * (0.0043)$$

where

R_s = Radius of sandpack in inches
R_w = Radius of well casing in inches
h_s = Height of sandpack in inches
h_w = Water depth in inches
0.0043 gallons (gal)/inch (in.)^3
Assumed filter pack porosity = 30 percent.

3. First round samples are to be collected from upgradient wells first, moving to downgradient wells under the assumption that upgradient wells will be less contaminated than downgradient wells. Results of first round analysis may mandate a change in sampling sequence.

Samples will always be collected in order of decreasing volatility (i.e., the samples to be analyzed for the volatile constituents should be collected first). Deliver the VOC sample to the vial by allowing the water to trickle down the inside wall of the vial at a rate no greater than approximately 100 mL/minute. Other samples may be delivered at a faster rate. Sampling rates will at no time exceed 1 L/minute. Procedures for each class of samples are contained in the site-specific Quality Assurance Project Plan.

When collecting samples for volatile analysis, care should be taken to prevent analyte loss by volatilization. The following procedures should be adhered to when collecting these samples:

- Avoid excessive aeration and agitation of sample.
- Fill vial so that a reverse meniscus is present by adjusting the flow rate from the sampling device.
- Place septum on vial so that the PTFE side is in contact with the sample. After the cap is on the bottle, check for air bubbles in the sample. If air bubbles are present, properly dispose of that sample and recollect the sample in the same vial or a new vial if prepreserved.
- Make sure vial is labeled and immediately transfer the vial to the cooler with ice.

Filtered and unfiltered samples will be taken for inorganics (metals) analyses, as appropriate. The samples will be filtered through an in-line 0.45-µM filter (preferred method), or by gravity through a 0.45-µM membrane placed in a filter funnel. Use forceps to place the membrane into the funnel and pour sample through funnel until appropriate volumes have been filtered.

If necessary, due to slow filtering, a peristaltic pump may be used to filter the sample through an in-line filter. Connect the pump to the generator, and attach tygon tubing to the bottom discharge valve on the bailer. Start pump and collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

— OR —

If a low flow pump is used collect the samples, filtered samples will be taken by installing a 0.45-µM filter in-line and pumping the water through the filter. Collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. If a flow-through cell is used to measure water quality parameters, collect samples before flow-through cell. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

Unfiltered samples will be collected by slowly pouring the sample water into the appropriate sample container, being careful not to agitate or cause bubbles to form. Do not overfill bottles. Make sure sample bottle is labeled and the cap is on tightly, then place the sample in cooler with ice immediately.

All samples will be delivered to the laboratory as soon as possible. If possible, samples will be shipped on the same day as they are collected. If samples must be retained due to weekend sampling (Friday through Sunday), the laboratory will be notified as to the time sensitive nature of the samples.

3.4 SAMPLING OF NON-AQUEOUS PHASE LIQUIDS

If NAPLs are detected in the well, a sample from all layers must be collected prior to any purging activities. NAPLs may be indicated by the presence of volatiles in the well headspace, and confirmed by the oil/water interface probe.

Collecting light non-aqueous phase liquid (LNAPL) will be accomplished using a transparent bailer with a double check valve. This bailer will be slowly lowered until the bottom of the bailer is 1-2 in. below the LNAPL-water interface, then slowly withdrawn. Verify that the interface was sampled by visual inspection of the bailer contents through the side of the bailer. Measure the thickness of the LNAPL in the bailer and note in the Field Logbook. Sample for laboratory analysis. An additional field verification may be performed by decanting the remainder of the contents of the bailer into a glass jar, adding a hydrophobic dye such as Sudan IV, or Redoil, shaking the sample and looking for coloration of NAPL. Alternate field tests are: examine the sample under ultraviolet light (many fluoresce), or allow the sample to stand overnight, and examine for interface and/or volatiles in the headspace the following day. Refer to the following sections on purging and sample collection for setup and general operation.

Collecting dense non-aqueous phase liquids (DNAPLs) will be accomplished using a transparent bailer with a double check valve. The bailer must be lowered very slowly to the bottom of the well and raised slowly out of the well in a controlled fashion. Sample for analysis as above. The same field check described above may be employed for DNAPL. Refer to the following sections on purging and sample collection for set up and general operation.

If NAPLs are present in the well, **and** a low-flow pump is to be used for purging and sampling, the well will be allowed to re-equilibrate prior to purging and sampling. This will be accomplished by allowing the well to stand undisturbed for at least 8 hours prior to purging and sample collection.

3.5 WELL PURGING GENERAL RULES

Water within the casing of a well will stagnate, de-gas, lose volatiles, possibly precipitate metals due to changes in redox potential, and may react with the screen and/or casing material. It is, therefore, necessary to purge a sufficient volume of this stagnant water from the well and/or casing to ensure that a representative sample of formation water can be obtained. Traditionally,

the volume of water to be purged was arbitrarily set at 3-5 equivalent volumes. Recent advances in sampling technologies have caused a re-thinking of such arbitrary purge volumes. It is, for this reason, that monitoring of select chemical and physical properties of the sample medium will be used instead of strict volumes to determine when a representative sample may be taken from a well.

Acceptable purge/sampling devices include: bailers, high-discharge submersible pumps (purge only), and variable speed, low-flow pumps that include both submersible pumps (purge and sample) and dedicated bladder pumps (purge and sampling). It is recommended to purge and sample at similar rates with one type device per well. An acceptable exception to this general rule is to use a high-discharge submersible pump to purge a deep, fast-recharging well, and a bailer to sample the same well.

Peristaltic, gas-lift, and centrifugal pumps can cause volatilization, produce high pressure differentials, and result in variability in the analysis of some analytes of interest. For this reason, these pumps should be used with caution and flow rate slowed to minimize volatilization.

To prevent groundwater from cascading down the sides of the screen into an open hole, thereby aerating the sample, purge rates will closely match recharge rates. If the static water level is within the casing, the initial purge rates may be set high enough to lower the water level to the top of the screen, then reduced to maintain that level and identify the well's recharge rate.

Purging will be accomplished with either a submersible pump, a low-flow (submersible or bladder) pump, or bailer. The choice of bailer or pump will be based on depth to water table, volume to be purged, and permeability of the aquifer. If the well recharges rapidly and/or has greater than 20 gal (estimated EV) to be purged, water may be removed with a submersible pump or a low-flow pump. If the well recharges slowly and/or has less than 20 gal to be purged, water will be removed with a bailer or a low-flow pump.

Purging will be accomplished with as minimal disturbance to the surrounding formation as possible.

Purge water will be containerized onsite until analysis of samples is completed. Based on sample results, accumulated purge water will be properly disposed of.

If the water level is within the screened interval and the well recharge rate is less than 0.1 L/minute, purge the well using a low-flow pump as follows:

1. Draw the water down to within 1 ft of the top of the pump.

2. Allow the well to recover.

3. Check and record field parameters.

4. Repeat Steps 1 through 3 then collect samples for metals analysis only[4].

5. Note the event in the Field Logbook, and report the problem to the Project Manager. If this extremely low recharge problem consistently occurs in a given well, the well may be considered for re-development and/or replacement.

6. If adjacent wells have elevated VOC levels, additional soil gas surveys will be considered in the vicinity of the low recharge well to help determine the need for replacement.

3.6 PURGING AND SAMPLING WITH BAILERS

Bailers may be used for both purging and sampling wells if: (1) the well recharge rate is less than 4 L/minute, (2) depth to the water table is less than 50 ft, and (4) less than 20 gal are to be purged (5 EV < 20 gal)[5].

When purging with a bailer, either a polyvinyl chloride, PTFE, or stainless steel bailer may be used. The bailer will be attached to either a spool of PTFE-coated stainless steel cable or polypropylene rope. If using cable, attach it to the bailer using stainless steel cable clamps. Thoroughly decontaminate the cable after each use, prior to rewinding cable onto spool. Cable clamps and raw cable ends may serve to trap contamination. Exercise particular caution in decontaminating these areas. If using rope, attach the rope to the bailer using a bowline knot, dispense the needed length (a few feet more than the well depth), and cut the remainder away; then, at the end opposite the bailer, make a slip knot and place it around the well casing or protective posts to prevent losing the bailer and rope down the well. The polypropylene rope will be not reused; it will be properly disposed of. Either type of bailer will be repeatedly lowered gently into the well until it fills with water, is removed, and the water discharged into an appropriate container until purging is complete. Care must be taken not to unduly agitate the water, as this tends to aerate the sample, increase turbidity, makes stabilization of required parameters difficult to achieve, and generally prolongs purging.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

4. Analyte losses due to volatilization in a drained well are too high for valid VOC sampling (McAlary and Barker 1987).
5. These numbers are based on the following assumptions: (1) In purging, it is preferable to remove water at approximately the recharge rate; (2) 4 L/minute is estimated as the approximate maximum rate at which water can be removed with a bailer from depths of 20-50 ft; and (3) 20 gal is estimated to be at the limit of the sampler's endurance, at which point fatigue and sloppiness of technique begin.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly. Refill bailer as needed.

If the controlled flow bottom discharge attachment is used for VOC sampling, attach it to the bottom of the bailer. Using the stopcock valve on the bailer to control the flow, and fill sample vials as described above in Section 3.3.

Remove check valve top and pour unfiltered sample into inorganics sample bottles.

Collect filtered samples as described in Section 3.3. Decontaminate bailer and cable.

3.7 PURGING WITH PUMP, SAMPLING WITH BAILER

If the recharge rate of the well is greater than 30 L/minute, or the water level is deeper than 50 ft, or more than 20 gal of purge water will be generated (5 EV > 20 gal), then purging and sampling may be accomplished using a submersible pump/bailer combination.

When purging with a pump, gradually lower the intake until it is submerged within the screened interval. Lower an electronic water level probe to the top of the screen (as determined from completion records) to the monitor water level, start pump, and slowly lower the pump as the water level continues to fall. Care should be exercised to lower the water column to the top of the screened interval (water level probe will stop beeping) but not below the top of the screen if possible. This will ensure that the stagnant layer has been removed, but should minimize the detrimental effects of over pumping the well. Secure hose(s) and/or power cord to casing and place discharge hose into the proper container, downhill and as far away from the well as possible. Determine and record the discharge rate.

Discharge rate = volume of container/time to fill container

The discharge rate will be established at approximately equal to or just greater than the well's recharge rate (determined from well development). If well development records are incomplete, recharge rate can be determined by monitoring the rise/fall of the water level within the casing as one purges the well. If the water level is static at a given pumping rate, but fluctuates up or down as pumping rate is decreased or increased, the pumping rate at which the water level is static is the recharge rate.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples,

sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with a double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles). Filtration of metals samples will be accomplished using either an in-line filter attached to the bottom of the bailer, or a funnel and appropriate filter (Section 3.3).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly, and fill sample containers as described in Section 3.6. Decontaminate bailer and cable in and decontaminate pump.

3.8 PURGING AND SAMPLING WITH LOW-FLOW PUMP

To obtain representative samples, subsurface disturbances should be kept to a minimum, thereby preventing sample alteration due to sampling actions. The reasoning behind the use of low-flow pumps to purge and sample monitoring wells is that these pumps minimize physical disturbance (turbulence) at the sampling point and chemical changes (aeration) in the medium. For these reasons, the low-flow pump is the preferred method for both purging and sampling in most cases. For the purposes of this SOP, “low-flow pumps” are defined as either dedicated bladder pumps or variable speed submersible pumps. Practical operational flow rates for these sampling devices range from 0.1 to 30 L/minute.

Low-flow pumps may be used for purging and sampling any well having recharge greater than 0.1 L/minute, which is the practical lower limit of pump performance. Below that pumping rate, pump inefficiencies and/or overheating may alter the physical and chemical properties of the sample. If the pump is continuously operated at sampling rates higher than the well recharge rate, the water level will be lowered in the well, possibly allowing aeration of the sample that is unacceptable sampling procedure. Low-flow pumps are suitable for sampling wells with recharge rates lower than 0.1 L/minute if precautions are taken to avoid aeration of the sample.

Low flow submersible pumps will be used as follows:

- Lower the pump into the well, slowly so as not to agitate the water, until the pump is at the mid-point of the screened interval or the mid-point of the water column if the static water table lies below the top of the screen.[6]

- Attach the pump's umbilical cord (which will consist of power cord and sampling tubing) to the protective casing, or lock the cord spool so that the pump cannot move vertically in the well during sampling.

- Lower the water level probe into the well behind the pump until it just touches water. This will allow the sampler to monitor the water level while purging and sampling, and prevent the inadvertent drying of the well.

- Begin purging at the pump's lowest setting, then gradually increase rate[7] until the pumping rate matches the aquifer recharge rate. **If the water level is above the top of the screen**, the pumping rate may be allowed to slightly exceed recharge rate, lowering the water level to no less than 1 ft above the screen, then reduced until it matches recharge rate and purging continued. **If the water level is below the top of the screen**, always keep the purge rate lower than well's recharge rate.

- Monitor stabilization parameters listed in Section 3.6 beginning immediately, using an in-line monitoring system. Record parameters regularly, at a rate of one set of parameters per each 1-3 liters of water removed from the well. When these parameters stabilize to within 10 percent over three consecutive readings, reduce[8] flow rate to 0.1 L/minute (if needed) and begin collecting VOC samples directly from the discharge line.

- If the well recharges at a rate less than 0.1 L/minute, purge until the water level is even with the top of the screen, allow the well to recover, and sample immediately.

- Remove and decontaminate water level probe and pump.

6. This assumes a 10-ft screened interval. If the screened interval is greater than 10 ft, multiple samples should be taken as follows:
   - If the screen is 10-12 ft, sample the canter of the water column, as outlined above.
   - If the screen is longer than 12 ft, and the water column is 10 ft or less, sample the center of the water column.
   - If the screen is longer than 12 ft, and the water column fills the screen, or extends above the screen, sample at 1/3 and 2/3 the height of the water column, or about every 6 ft.
7. Some sources indicate that the pumping rate should not exceed 1 L/minute, with 0.5 L/minute being preferable. The optimal purge rate is highly aquifer dependent, and may range from less than 0.5 L/minute to greater than 10 L/minute. The purge rate for a given well will, therefore, be a field decision, based on well development, purge, and sampling records rather than SOP mandate.
8. Sampling should occur at the same rate as purging as long as aeration of sample does not occur.

4. MAINTENANCE

Refer to manufacturer's requirements for maintenance of pumps and generators.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for appropriate personal protective equipment.

6. REFERENCES

McAlary, T.A. and J.F. Barker. 1987. Volatilization Losses of Organics During Groundwater Sampling From Low Permeability Materials, in Groundwater Monitoring Review. Fall.

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Standard Operating Procedure No. 016
for
Surface Water, Groundwater, and Soil/Sediment Field Logbooks

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for recording surface water, groundwater, soil/sediment sampling information, instrument calibration data, and data from hydrologic testing in the field logbooks. Acceptable field logbooks are: bound, unprinted books such as a surveyor's field book, or a federal supply service No. 7530-00-222-3525 record book (or equivalent); or they may be company-proprietary, pre-printed forms bound into a field logbook. Example forms are provided herein. Alternate, equivalent forms are acceptable.

2. MATERIALS

The following material may be required: applicable field logbook and indelible ink pen.

3. PROCEDURE

Information pertinent to soil/sediment, groundwater, or surface water sampling will be recorded in the appropriate logbook. Each page/form of the logbook will be consecutively numbered. Entries will be made in indelible ink. Corrections will consist of line-out deletions that are initialed and dated. If using carbon paper or self-duplicating forms, before entering data in logbook, insert a sheet protector between form sets to isolate first blank form from remaining forms.

3.1 SOIL/SEDIMENT LOGBOOK (Requires Figures SOP016-1 and SOP016-3)

3.1.1 Field Parameter Form (Items on Figures SOP016-1 and SOP016-2)

1. HIGH CONCENTRATION EXPECTED?: Answer "Yes" or "No."

2. HIGH HAZARD?: Answer "Yes" or "No."

3. SITE: Record the complete name of the site.

4. AREA: Record the area designation of the sample site.

5. INST CODE: Record the 2-letter installation code appropriate for the installation or site. Correct abbreviations can be found on Pages 3-6 of the IRDMS User's Guide for chemical data entry.

6. FILE NAME: Record "CSO" for a soil sample or "CSE" for a sediment sample.

7. SITE TYPE: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry. This entry must match the Site Type on the map file form.

8. SITE ID: Record a code up to 10 characters or numbers which is unique to the site.

9. FIELD SAMPLE NUMBER: Record a code specific for the sample.

10. DATE: Enter the date the sample was taken.

11. TIME: Enter the time (12-hour or 24-hour clock acceptable as long as internally consistent) the sample was taken.

12. AM PM: Circle "AM" or "PM" to designate morning or afternoon (12-hour clock).

13. SAMPLE PROG: Record "GQA" (Groundwater Quality Assessment) or other appropriate sample program.

14. DEPTH (TOP): Record the total depth sampled.

15. DEPTH INTERVAL: Record the intervals at which the plug will be sampled.

16. UNITS: Record the units of depth (feet, meters)

17. SAMPLE MEASUREMENTS: Check the appropriate sampling method.

18. CHK: Check off each container released to a laboratory.

19. ANALYSIS: Record the type of analysis to be performed on each sample container.

20. SAMPLE CONTAINER: Record the sample container type and size.

21. NO.: Record the number of containers.

22. REMARKS: Record any remarks about the sample

23. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: Record the total number of containers.

24. SITE DESCRIPTION: Describe the location where the sample was collected.

25. SAMPLE FORM: Record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System.

26. COLOR: Record the color of the sample as determined from standard Munsell Color Charts.

27. ODOR: Record the odor of the sample or "none." See SOP No. 001 Section 5.

28. PID (HNu): Record the measured PID (HNu) values.

29. UNUSUAL FEATURES: Record anything unusual about the site or sample.

30. WEATHER/TEMPERATURE: Record the weather and temperature.

31. SAMPLER: Record your name.

3.1.2 Map File Form (Figure SOP016-3)

1. The map file logbook form will be located on the reverse of the field parameter logbook form, or on an adjoining page of the field logbook (if level book is used).

2. SITE ID: Record the Site ID from the field parameter form.

3. POINTER: Record the field sample number for the sample being pointed to.

4. DESCRIPTION/MEASUREMENTS: Describe the location where the sample was taken, along with distances to landmarks.

5. SKETCH/DIMENSIONS: Diagram the surroundings and record the distances to landmarks.

6. MAP REFERENCE: Record which U.S. Geological Survey Quad Map references the site.

7. COORDINATE DEFINITION: Write the compass directions the X- and Y-Coordinates of the map run.

8. COORDINATE SYSTEM: Write "UTM" (Universal Transverse Mercator).

9. SOURCE: Record the 1-digit code representing the Map Reference.

10. ACCURACY: Give units (e.g., write "1-M" for 1 meter).

11. X-COORDINATE: Record the X-Coordinate of the sample site location.

12. Y-COORDINATE: Record the Y-Coordinate of the sample site location.

13. UNITS: Record the unit's map sections are measured in.

14. ELEVATION REFERENCE: Record whether topography was determined from a map or a topographical survey.

15. ELEVATION SOURCE: Record the 1-digit code representing the elevation reference.

16. ACCURACY: Record the accuracy of the map or survey providing the topographical information.

17. ELEVATION: Record the elevation of the sampling site.

18. UNITS: Write the units in which the elevation is recorded.

19. SAMPLER: Write your name.

3.2 SURFACE WATER LOGBOOK (Requires Figures SOP016-2 and SOP016-3)

3.2.1 Field Parameter Form (Items Unique to Figure SOP016-3)

1. CAL REF: Record the calibration reference for the pH meter.

2. pH: Record the pH of the sample.

3. TEMP: Record the temperature of the sample in degrees Celsius.

4. COND: Record the conductivity of the water.

5. For all other sections, see Section 3.2.1.

3.3 GROUNDWATER SAMPLING LOGBOOK (Requires Figures SOP016-2, SOP016-3, and SOP016-4)

3.3.1 Field Parameter Form (Items on Figure SOP016-4)

1. WELL NO. OR ID: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry.

2. SAMPLE NO.: Record the reference number of the sample.

3. WELL/SITE DESCRIPTION: Describe the location where the sample was taken, along with distances to landmarks.

4. X-COORD and Y-COORD: Record the survey coordinates for the sampling site.

5. ELEV: Record the elevation where the sample was taken.

6. UNITS: Record the units the elevation was recorded in.

7. DATE: Record the date in the form MM/DD/YY.

8. TIME: Record the time, including a designation of AM or PM.

9. AIR TEMP.: Record the air temperature, including a designation of C or F (Celsius or Fahrenheit).

10. WELL DEPTH: Record the depth of the well in feet and inches.

11. CASING HT.: Record the height of the casing in feet and inches.

12. WATER DEPTH: Record the depth (underground) of the water in feet and inches.

13. WELL DIAMETER: Record the diameter of the well in inches.

14. WATER COLUMN HEIGHT: Record the height of the water column in feet and inches.

15. SANDPACK DIAM.: Record the diameter of the sandpack. Generally, this will be the same as the bore diameter.

16. EQUIVALENT VOLUME OF STANDING WATER: Use one of the following equations, to determine one equivalent volume (EV):

    1 EV = Volume in casing + volume in saturated sand pack. Or to restate:

    $1\ EV = (BR_w^2 h_w + 0.30B(R_s^2 - R_w^2)h_s) * (0.0043)$

    where
    R_s = Radius of sandpack in inches
    R_w = Radius of well casing in inches
    h_s = Height of sandpack in inches
    h_w = Water depth in inches

    $0.0043 = gal/in.^3$
    and filter pack porosity is assumed as 30 percent

    — OR —

    Volume in casing = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_c^2)(W_h)$

    where
    R_c = Radius of casing in inches
    W_h = Water column height in feet

    Vol. in sandpack = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_b^2 - R_c^2)(W_h)(0.30)$

    (if W_h is less than the length of the sandpack),

— PLUS —

Vol. in sandpack = $(0.0043 \text{ gal/in.}^3)(B)(12 \text{ in./ft})(R_b^2 - R_c^2)(S_h)(0.30)$

(if W_h is greater than the length of the sandpack).

where
R_b = Radius of the borehole
S_h = Length of the sandpack.

Show this calculation in the comments section.

17. VOLUME OF BAILER OR PUMP RATE: Record bailer volume or pump rate.

18. TOTAL NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers required to remove 3 equivalent volumes (EV) of water from the well or the total purge time and volume as applicable.

19. WELL WENT DRY? Write "YES" OR "NO."

20. NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers or pump time which made the well go dry.

21. VOLUME REMOVED: Record the volume of water (gal) removed before the well went dry.

22. RECOVERY TIME: Record the time required for the well to refill.

23. PURGE AGAIN?: Answer "YES" or "NO."

24. TOTAL VOL. REMOVED: Record the total volume of water (in gal) removed from the well.

25. CAL REF.: Record the calibration reference for the pH meter.

26. TIME: Record time started (INITIAL T[0]), 2 times DURING the sampling and the time sampling ended (FINAL).

27. pH: Record the pH at start of sampling (INITIAL), twice DURING the sampling and at the end of sampling (FINAL).

28. TEMP: Record the water temperature (Celsius) at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

29. COND: Record the conductivity of the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

30. D.O.: Record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

31. TURBIDITY: Record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

32. ORD: Record the oxidation/reduction (RedOx) potential of the water sample at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

33. HEAD SPACE: Record any positive readings from organic vapor meter reading taken in well headspace prior to sampling.

34. NAPL: Record the presence and thickness of any non-aqueous phase liquids (light or dense)

35. COMMENTS: Record any pertinent information not already covered in the form.

36. SIGNATURE: Sign the form.

3.4 FIELD CALIBRATION FORMS (Maintained as a separate logbook, or incorporated into sampling logbooks)

3.4.1 Items on Figure SOP016-5

1. Record time and date of calibration. Note whether 12- or 24-hour clock was used.

2. Record calibration standard reference number.

3. Record meter I.D. number

4. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line.

5. Record value of reference standard (as required).

6. COMMENTS: Record any pertinent information not already covered on form.

7. SIGNATURE: Sign form.

3.5 GROUNDWATER HYDROLOGY TESTS LOGBOOK (Must include Figures SOP016-6 and SOP016-7 and/or SOP016-8, OR SOP016-9 or SOP016-10)

3.5.1 Field Permeability Test Data Sheet (Items on Figures SOP016-6)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

4. PROJECT NO.: Record the contractor assigned project number or the contract number.

5. LOCATION: Specific location

6. CLIENT: Agency or company with the contract under which the work is being performed.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

9. TEST TYPE: Short description of the type of test to be performed.

10. RISING/FALLING HEAD WITH SLUG: Check if the test involved the insertion/removal of and inert object.

11. RISING/FALLING HEAD WITHOUT SLUG: Check if the test involved the addition/removal of a quantity of water.

12. START DATE: Date on which the test was begun.

13. CLOCK TIME: Time each datum (depth to groundwater level) is collected. Note whether 12- or 24-hour clock was used.

14. ELAPSED TIME: Time since the last datum was collected.

15. DEPTH TO GWL (ft): Depth to the top of the groundwater table (Groundwater Level) as measured by manual methods.

16. REC. (ft): Water level as reported by transducer/datalogger (this is the depth of water above the transducer.

17. TIME: Time the discharge rate check was begun (addition or removal of water method). Note whether 12- or 24-hour clock was used.

18. FLOW METER (Addition or removal of water method): The amount of water added or removed as registered by the flowmeter, in gal of liters.

19. DISCHARGE RATE: Flowmeter reading divided by time interval (gal/min or liters/min).

20. SIGNATURE: The person completing this form must sign the form at the end of the test.

21. DATE: Date the form was signed.

3.5.2 Groundwater Levels – Single Well (Items on Figure SOP016-7)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

Well Data

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: Remedial investigation wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATE: Date of each water level reading

16. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

17. ELAPSED TIME: Time since test was begun.

18. DEPTH TO WATER: Measured depth to the groundwater table.

19. WATER ELEVATION: Elevation of the top of the groundwater table (use datum listed above).

20. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

21. TAPE NO.: The unique identification number of the traceable standard tape used to calibrate the measuring device.

22. WELL STATUS: Condition of the well at the time of measuring (see abbreviation key at the bottom of the data sheet).

23. REMARKS: Any additional pertinent comments not specifically required above.

24. INITIALS: Initials of person completing this data entry.

25. ABBREVIATION KEYS: Self explanatory.

26. SIGNATURE: The person completing this form must sign the form at the end of the test.

27. DATE: Date the form was signed.

3.5.3 Groundwater Levels – Single Well (Items on Figure SOP016-8)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATALOGGER: This section is record of pertinent datalogger information.

16. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

17. MODEL: Enter the model number of the datalogger.

18. S/N: Enter the serial number of this datalogger.

19. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

20. TRANSDUCER: This section is a listing of pertinent information about the transducer used.

21. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

22. MODEL: Enter the model number of the transducer.

23. S/N: Enter the serial number of this transducer.

24. INPUT/UNITS: What are the units this transducer uses?

25. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

26. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer. (Usually in psi, or kpa).

27. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

28. VOLUME WATER ADDED/REMOVED: (Applicable if inert object insertion/removal method was not employed.) Record the volume of water added to or removed from the well.

29. DISCHARGE RATE: If z (above) is filled, enter the rate at which this water was added or removed.

30. INITIAL WATER LEVEL (ft): Enter the water level in the well at the beginning of the test.

31. PRESSURE TRANSDUCER SUBMERGENCE: Record the depth to which the transducer is submerged at the beginning of the test and the depth to the transducer at the end if the test. All depths will be recorded to the nearest 0.01 ft.

32. TIME: Record the time the test is begun and ended. Note whether 12- or 24-hour clock was used.

33. OBSERVED CHANGES IN ADJACENT WELLS: Note any changes in water levels in nearby wells.

34. RESULTS RECORDED ON DISKETTE #: Tracking number of the diskette on which these data are archived.

35. DISKETTE FILE NAME: Name of the file(s).

36. SIGNATURE: The person completing this form must sign the form at the end of the test

37. DATE: Date the form was signed.

3.6 GROUNDWATER LEVELS – MULTIPLE WELLS (Items on Figure SOP016-9)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

5. LOCATION: Specific location.

6. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

7. CLIENT: Agency with the contract under which the work is being performed.

8. REMARKS: Any pertinent observations not specifically required above.

9. WELL: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

10. DATE: Date this measurement was made.

11. TIME: Time this measurement was made. Note whether 12- or 24-hour clock was used.

12. DEPTH TO WATER: Depth from MP to top of groundwater table.

13. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

14. MP ELEV.: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

15. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

16. REMARKS/MP: Describe the location and nature of the measuring point.

17. INITIALS: Initials of the person completing this form.

18. ABBREVIATION KEYS: Self explanatory.

19. SIGNATURE: The person completing this form must sign the form at the end of the test.

20. DATE: Date the form was signed.

3.7 GROUNDWATER LEVELS – DATALOGGERS (Items on Figure SOP016-10)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019, Section 3.4).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

DATALOGGER (This section is a record of pertinent datalogger information)

15. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

16. MODEL: Enter the model number of the datalogger.

17. S/N: Enter the serial number of this datalogger.

18. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

TRANSDUCER (This section is a listing of pertinent information about the transducer used)

19. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

20. MODEL: Enter the model number of the transducer.

21. S/N: Enter the serial number of this transducer.

22. INPUT/UNITS: What are the units this transducer uses?

23. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

24. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer (usually in psi, or kpa).

25. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

26. DATE: Date of each water level reading

27. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

28. LOGGING TIME INTERVAL: Time since test was begun.

29. WL FEET BELOW MP: Measured depth to the groundwater table from measuring point.

30. SUBMERGENCE: Depth of water above the transducer.

31. MEAS.METHOD: What device/method was used to measure the water level.

32. TAPE NO.: Record the tape identification number.

33. TRANSDUCER MOVED?: Was the transducer moved since the last water level reading?

34. REMARKS: Any pertinent remarks not otherwise specified.

35. INITIALS:

DATA TRANSFER TO DISKETTE:

36. DATE: Date data were archived onto diskette.

37. TIME: Time stamp the computer assigns the data file.

38. FILE NAME: Name assigned the data file.

39. SOFTWARE USED FOR TRANSFER: Any special software, or computer operating system used to write the files to diskette. NOTE: If a "shareware" archiver which compresses files was used, and the archived file is not self-extracting, a copy of the unarchive program should be copied onto the diskette also.

40. OUTPUT FORMAT: What is the format of the output file? (DOS, UNIX, Binary, Compressed?)

41. INITIALS: Initials of the person who copied the data to diskette.

42. ABBREVIATION KEY: Self-explanatory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency. 1984. User's Guide to the Contract Laboratory Program. July.

FIGURE SOP016-1
FIELD PARAMETER LOGBOOK
SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

INSTALLATION/SITE _____ AREA _____

INST CODE FILE NAME _____

SITE TYPE SITE ID
FIELD SAMPLE NUMBER

DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.

DEPTH (TOP) DEPTH INTERVAL UNIT _____

SAMPLING METHOD:

SPLIT SPOON AUGER SHELBY TUBE SCOOP OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION:_____
SAMPLE FORM _____ COLOR _____ ODOR _____
PID (HNu) _____ UNUSUAL FEATURES _____
_____
WEATHER/TEMPERATURE
SAMPLER

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

FIGURE SOP016-2
FIELD PARAMETER LOGBOOK
GROUNDWATER AND SURFACE WATER SAMPLES

INSTALLATION/SITE AREA
INST CODE FILE NAME SITE TYPE
SITE ID FIELD SAMPLE NUMBER
DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.
DEPTH (TOP) DEPTH INTERVAL UNITS

SAMPLING MEASUREMENTS

CAL REF.	pH	TEMPERATURE C	CONDUCTIVITY	OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION
SAMPLING METHOD
SAMPLE FORM COLOR ODOR
PID (HNu)
UNUSUAL FEATURES
WEATHER/TEMPERATURE _____ SAMPLER _____

FIGURE SOP016-3
MAP FILE LOGBOOK

SITE ID POINTER _____
DESCRIPTION/MEASUREMENTS
SKETCH/DIMENSIONS:

MAP REFERENCE
COORDINATE DEFINITION (X is Y is)
COORDINATE SYSTEM SOURCE ACCURACY
X-COORDINATE Y-COORDINATE UNITS
ELEVATION REFERENCE
ELEVATION SOURCE ACCURACY ELEVATION
UNITS

SAMPLER

FIGURE SOP016-4
MAP FILE AND PURGING LOGBOOK
GROUNDWATER SAMPLES

WELL COORD. OR ID SAMPLE NO._____
WELL/SITE
DESCRIPTION_____
_____
X-COORD. Y-COORD. _____ ELEV. UNITS
DATE ____/____/____ TIME AIR TEMP.

WELL DEPTH _____ ft _____ in. CASING HT. _____ ft _____ in.
WATER DEPTH _____ ft _____ in. WELL DIAMETER _____ in.
WATER COLUMN HEIGHT _____ ft _____ in. SANDPACK DIAM. _____ in.
EQUIVALENT VOLUME OF STANDING WATER _____ (gal) (L)
VOLUME OF BAILER _____ (gal) (L) or PUMP RATE _____ (gpm) (lpm)
TOTAL NO. OF BAILERS (5 EV)_____ or PUMP TIME _____ MIN.
WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME_____MIN
VOL. REMOVED _____ (gal) (L) RECOVERY TIME _____ MIN
PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (gal) (L)

Date and Time	Quantity Removed	Time Required	pH	Cond	Temp	ORD	Turb	DO	Character of water (color/ clarity/odor/partic.)
(before)									
(during)									
(during)									
(during									
(after)									

COMMENTS:_____
_____
_____
_____

_____
SIGNATURE

FIGURE SOP016-5
FIELD CALIBRATION: pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, OXIDATION-REDUCTION POTENTIAL, AND DISSOLVED OXYGEN METERS

INITIAL CALIBRATION	FINAL CALIBRATION
DATE:	DATE:
TIME:	TIME:

pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

pH STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
7.0			
10.0			
4.0			

CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

COND. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

TEMPERATURE METER CALIBRATION

METER ID _____

TEMP. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
ICE WATER			
BOILING WATER			
OTHER			

FIGURE SOP016-5 *(continued)*

TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

COMMENTS:_____

_____

_____

_____

_____

SIGNATURE

FIGURE SOP016-6
FIELD PERMEABILITY TEST DATA SHEET

Contractor: **Seq. #** /

Project Name:	Project #:
Location:	Client:
Party Chief:	Contractor:
Observation Well:	
Test Type: Rising/Falling Head w/Slug	Rising/Falling Head w/out Slug

Start Date:								Discharge Rate		
Clock Time	Elapsed Time (min)	Depth to GWL (ft)	Rec (ft)	Clock Time	Elapsed Time	Depth to GWL (ft)	Rec (ft)	Time	Flow Meter	Discharge Rate

Signature: _____ **Date:** _____

FIGURE SOP016-7
GROUNDWATER LEVELS – SINGLE WELL

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:
Well No.: Site: Area:
Site: Area:
Area:

up (+)/down (-) from: Datum = MSL or:
Datum = MSL or:

Measuring Point Description:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:

Transducer: Manufacturer: Model: S/N:
Input/Units: Range:

Calibration:
Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v) / (mv)

Volume Water Added/Removed:
Discharge Rate:
Initial Water Level (ft):

Pressure Transducer Submergence
Initial (ft): Final(ft): Time:Start: End:
Observed Changes in Adjacent Wells:

Results Recorded on Diskette #:
Diskette File Name:

Signature: _____ **Date:** _____

FIGURE SOP016-8
GROUNDWATER LEVELS – MULTIPLE WELLS

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:

Measuring Point Description:
Remarks:
Well No.:
Site: Area: up (+)/down (-) from:
up (+)/down (-) from:
up (+)/down (-) from:
Datum = MSL or:

Date	Time	Elapsed Time	Depth to Water	Water Elevation	Meas. Meth.	Tape No.	Well Status	Remarks	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

Signature: _____ **Date:** _____

FIGURE SOP016-8 *(continued)*

Contractor: **Seq. #** /

Project No.:
Project Name: Location:
Field Party Chief: Client:

Well	Date	Time	Depth to Water	Stickup	MP Elev.	Meas. Meth.	Tape No.	Remarks/MP	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

FIGURE SOP016-9
GROUNDWATER LEVELS DATALOGGERS

Contractor
Project No.: **Well No.:**
Project Name: **Site:**
Field Party Chief: **Area:**

WELL DATA: Stickup: **(ft)** **up (+)/down (-) from:**
MP Elevation: **Datum = MSL or:**

Measuring Point Description:
Remarks:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:
Transducer: Manufacturer: Model: S/N:
Input/Units: Range:
Calibration: Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v)

Logging	Date	Time	Logging Time Interval	WL, ft Below MP	Submergence (logger reading)	Meas. Method	Tape No.	Well Status	Transducer Moved	Remarks	Initials
Start											
Stop											
Start											
Stop											

Data Transfer to Disk

Date	Time	File Name	Software Used for Transfer	Output Format	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently
NP = Nearby well pumping
NRP = Nearby well recently pumped
X = Obstructed

Signature **Date**

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Standard Operating Procedure No. 019
for
Monitoring Well Installation

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Revision 0
December 2014

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CONTENTS

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1. SCOPE AND APPLICATION

The installation of monitoring wells is contingent upon the existing conditions at the project site. The purpose of this Standard Operating Procedure is to delineate the quality control measures required to ensure the accurate installation of monitoring wells. The applicable Work Plan should be consulted for specific installation instructions. The term "monitoring wells," as used herein, is defined to denote any environmental sampling well. An example well log form is provided in Appendix A. Alternate, equivalent forms are acceptable.

2. MATERIALS

2.1 DRILLING EQUIPMENT

The following drilling equipment may be required:

- Appropriately sized drill adequately equipped with augers, bits, drill stem, etc.
- Steam cleaner and water obtained from approved source for decontaminating drilling equipment.
- Photoionization Detector: Microtip HL-200 (or equivalent)
- Water level indicator
- Weighted steel tape measure
- Lower explosive limit – oxygen monitor
- Steel drums for intrusion derived wastes (drill cuttings, contaminated personal protective equipment, decontamination solutions, etc.)
- Source of approved water
- Heavy plastic sheeting
- Sorbent pads and/or log.

2.2 WELL INSTALLATION MATERIALS[1]

The following well installation materials may be required:

- Well screen:[2]
  - — Polyvinyl chloride (PVC): JOHNSON (or equivalent); PVC 0.010 slot; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with American Society for Testing and Materials (ASTM) D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); stainless steel 0.010 slot; 304 stainless steel[3]; ASTM F480 flush threads; cleaned, wrapped, and heat sealed by manufacturer.
- Riser pipe:
  - — PVC: JOHNSON (or equivalent); STD; PVC; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); Schedule 5; 304 stainless steel; ASTM Type A312 material; 4-in. diameter; cleaned, wrapped, and heat sealed by manufacturer.
- Plugs/caps: JOHNSON (or equivalent); standard PVC or stainless steel.
- Filter pack: MORIE, 100 well gravel (or equivalent). NOTE: Final gradation may vary as a function of the gradation of the formation.[2]
- Fine Ottawa sand.
- Bentonite seal: BAROID, bentonite pellets (3/8-in. diameter)
- Cement: Type II Portland Cement (table below).

1. Technical information on all installed materials (screens, riser pipe, filter pack, bentonite, cement, etc.) and representative samples of the proposed filter pack, bentonite powder, and bentonite pellets will be supplied to the Project Manager.
2. Well screen slot size and filter pack gradation will be determined from sieve analysis of aquifer materials. Screen and casing material type will be determined based on field tests of groundwater chemistry and contaminants.
3. Unless the sum of Cl-, F-, and Br- is >1,000 ppm, in which case Type 316 should be used.

Cement Type	Special Characteristics	Recommended Usage
I	No special properties	General use as grout mix or cement plug (if sulfates <250 ppm), surface pad.
IA	Air-entraining Type I (Note that air entrainment properties can be achieved by chemical admixtures)	Air entrainment gives cement greater freeze-thaw resistance. Recommended for surface pads.
II	Moderate sulfate resistance, low heat of hydration	General use as grout mix or cement plug where groundwater sulfate >250 ppm and <1,500 ppm, surface pad.
IIA	Air-entraining Type II	See Type IA.
III	High early strength, high heat of hydration	Elevated temperature can damage well casing and fracture grout/cement plugs. NOT RECOMMENDED.
IIIA	Air-entraining Type III	NOT RECOMMENDED.
IV	Low heat of hydration	General use as grout mix or cement plug preferred type for well abandonment to ensure intact grout/cement plug.
V	High Sulfate resistance	Use when groundwater sulfate levels >1,500 ppm.

- Bentonite powder: BAROID, Aquagel Gold Seal.
- Steel protective casing: BRAINARD-KILMAN (or equivalent) zinc-plated steel, lockable, painted.[4]
- Geotextile: MIRAFI (or equivalent); GTF 130; non-woven; 4 oz.
- Coarse (blanket) gravel: Crushed stone aggregate.
- Containers for purged water, as required.
- Submersible pump or bailer of appropriate capacity, and surge block sized to fit well.
- Hach DREL 2000 portable laboratory (or equivalent).
- Conductivity, pH, oxidation-reduction potential (ORP), turbidity, dissolved oxygen, and temperature meters.
- Electric well sounder and measuring tape.
- Portland Type II cement (see previous table).
- Steel Posts (pickets), painted (see footnote).

4. All painted components (protector casing, steel pickets) will be painted high-visibility orange and allowed to dry completely prior to being brought onsite.

2.3 DOCUMENTATION

The following document may be provided:

- Copy of appropriate Work Plan
- Copy of approved Health and Safety Plan
- Copies of well and excavation permits
- Boring log forms
- Well completion diagram form
- Well development form.

2.4 GEOLOGIST'S PERSONAL EQUIPMENT

The following equipment may be required for the geologist:

- 10X handlens
- Unified Soil classification System chart
- Munsell color chart
- Sieve set (Keck model SS-81 or equivalent)
- Personal protective equipment as required by the Health and Safety Plan.

3. PROCEDURE

3.1 MATERIALS APPROVAL

Water sources for drilling, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the drilling equipment. Information required for the water source includes: water source, manufacturer/ owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analyses over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives (bentonite) will be the only drilling fluid additive allowed, and its use must be approved by the Project Manager prior to the arrival of the drilling equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product description, and intended use for the product.

Granular Filter Pack material must be approved by the Project Manager prior to drilling. A 1-pint representative sample must be supplied to the Project Manager. Information required includes: lithology, grain size distribution, brand name, source, processing method, and slot size of intended screen.

Portland Type II cement will be used for grout (see previous table).

3.2 DRILLING

The objective of the selected drilling technique is to ensure that the drilling method provides representative data while minimizing subsurface contamination, cross-contamination of aquifers, and drilling costs. The preferred drilling method is with a hollow-stem auger. Other drilling methods[5] are approved as conditions warrant, and will not require variances be issued by the U.S. Environmental Protection Agency. The method used at a specific site will be proposed in the work plan and evaluated by the Project Manager. Any drilling method not listed herein will require approval on a case by case basis by the U.S. Environmental Protection Agency.

A Site Geologist will be present during all well drilling and installation activities and will fully characterize all tasks performed in support of these activities into the monitoring well logbook. The Site Geologist will be responsible at only one operating rig for the logging of samples, monitoring of drilling operations, recording of water losses/gains and groundwater data, preparing the boring logs and well diagrams, and recording the well installation procedures of the rig. The Site Geologist will have onsite sufficient equipment in operable condition to perform efficiently his/her duties as outlined in the contractual documents. Items in the possession of each Site Geologist will include the approved Health and Safety Plan, this Standard Operating Procedure, a hand lens (10X), a standard color chart, grain-size chart, and a weighted (with steel or iron) steel tape long enough to measure the deepest well, heavy enough to reach that depth, and small enough to fit readily within the annulus between the well and drill casing. The Site Geologist will also have onsite, a water level measuring device, preferably electrical.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole drilling equipment. Additives containing either lead or copper will not be allowed. In addition, polychlorinated biphenyls will not be permitted in hydraulic fluids or other fluids used in the drilling rig, pumps, and field equipment/vehicles.

5. If the design depth of the well is <100 ft, open, hollow-stem augers will be used to drill the well unless "running sands" preclude the use of open augers. In that case, an inert "knockout" plug may be used in the bottom of the auger string. This plug will be driven out of the augers and left at the bottom of the hole when the well is installed.

   If the design depth of the well is >100 ft, rotary drilling methods may be used to install wells. The following drill fluids and methods are approved in the order listed: (1) rotary drilling with water from an approved source as drilling fluid (clays from the formations will tend thicken the fluid and coat the walls of the borehole and this is acceptable); (2) rotary drilling with water as a fluid, advancing a temporary casing with the bit to maintain an open hole; and (3) mud rotary using water with additives as drill fluid. Due to the potential for aquifer contamination and plugging, mud rotary drilling is not recommended for monitoring wells. If, however, "running sands" are encountered and the aquifer is expected to have a relatively high flow rate, then mud rotary is considered an approved method. Pure sodium bentonite is the only approved additive. Mud rotary drilling must be halted at the last aquitard above the target aquifer. Casing must be set, all bentonite-bearing fluids flushed from the hole and drill rig, and drilling may be resumed using water only as the drill fluid until the target depth is reached.

Surface runoff or other fluids will not be allowed to enter any boring or well during or after drilling/construction.

Antifreeze used to keep equipment from freezing will not contain rust inhibitors and sealants. Antifreeze is prohibited in areas in contact with drilling fluid. The ground surface at the well site will be protected from possible coolant, fuel, and hydraulic fluid spills and/or leakage by placement of plastic sheeting with raised edges, draining into a lined catch basin large enough to contain spills and/or leakage from motors, radiators, or vehicle tanks. Sorbent pillows will be placed to catch obvious leaks from the drill rig. Sorbent logs may be used instead of, or in conjunction with, a lined catch basin to contain spills.

An accurate measurement of the water level will be made upon encountering water in the borehole and later upon stabilization. Levels will be periodically checked throughout the course of drilling. Any unusual change in the water level in the hole, such as a sudden rise of a few inches may indicate artesian pressure in a confined aquifer, will be the basis for cessation of drilling. The geologist will immediately contact the Project Manager[6]. Particular attention for such water level changes will be given after penetrating any clay or silt bed, regardless of thickness, which has the potential to act as a confining layer.

Anticipated depths of wells are given in well specific work plans. In case the previously defined criteria have not been met before the depth range for a given hole is reached, the geologist will stop the drilling and confer with the Project Manager. The current boring conditions (depth, nature of the stratigraphic unit, and water table depth) will be compared to those of other wells nearby to decide to continue drilling or to terminate and complete the well.

If the well is to be installed in the surficial aquifer, drilling will be terminated before penetrating the basal aquitard. The basal aquitard is defined as the first 2 ft-thick clay below the water table, or below 5 ft in the case of a shallow aquifer.

If the well is to be installed in a lower, confined aquifer:

- Penetrations of aquifers located lower than the water table aquifer will be limited to avoid cross-contamination.

- Placement of new upper confined aquifer wells will be initially limited to those areas where contamination has been confirmed.

- The location of upper confined aquifer wells will be based upon the findings of the water table aquifer investigation. Areas of known contamination will be targeted for installing upper confined aquifer wells for the purposes of delineating vertical contamination.

6. The contract technical oversight will also be contacted for guidance.

- Where possible, upper-confined aquifer wells will be located such that they afford triangulation with other wells within the same aquifer to allow for a determination of groundwater flow direction.

- Some upper-confined aquifer wells will be installed approximately 10-15 ft from water table wells to enable the accurate assessment of vertical hydraulic gradients. If the direction of groundwater flow is known, wells within a group will be located sidegradient of each other.

- The boring will be advanced until the base of the surficial aquifer is reached (Section 3.2).

- An outer, surface casing will be set 2-5 ft into the confining layer to minimize the potential for cross-contamination from the unconfined aquifer during drilling activities.

- The surface casing will be driven into the confining bed and grouted into place. Grout will be tremied into the annulus around the outside of the casing to within 5 ft of the ground surface. A grout plug at least 2 ft thick will be tremied into the bottom of the surface casing. The grout will be permitted to cure for 24 hours. All drilling fluids within the surface calling will then be removed, and the casing will be flushed with clean potable water.

- The drilling equipment will be decontaminated, a smaller bit or auger selected, and the hole will be continued through the grout plug into the confined aquifer.

- If deeper aquifers are to be screened, repeat preceding steps until total depth is reached.

If dense non-aqueous phase liquid (DNAPL) contamination is detected during drilling, the well will be terminated and completed at the base of the aquifer. Drilling will not continue through the confining unit.

Stainless steel screens will be used in DNAPL wells. Screen size selection will be according to criteria set forth in Section 3.4. The formation grain size will be multiplied by the higher factor (6) to determine filter pack grain size. This will ensure that the filter pack is sufficiently coarse to permit DNAPL to pass freely from the formation into the coarser filter pack, then into the open well (Cohen and Mercer 1993).

DNAPL sampling cups are prohibited. The well screen will be capped, and set 0.3 ft (0.5 ft max.) into the top of the confining bed and rest on the bottom of the hole or bentonite backfill (if used). No sand will be placed below the screen. The remainder of the well installation and completion will be accomplished according to Section 3.4.

3.3 LOGGING

All borings for monitoring wells will be logged by a geologist. Logs will be recorded in a field logbook and/or a boring log. If the information is recorded in a logbook, it will be transferred to Boring Log Forms on a daily basis. Field notes are to include, as a minimum:

- Boring number
- Material description (as discussed below)
- Weather conditions
- Evidence of contamination
- Water conditions (including measured water levels)
- Daily drilling footage and quantities (for billing purposes)
- Notations on man-placed materials
- Drilling method and borehole diameter
- Any deviations from established field plans
- Blow counts for standard penetration tests
- Core and split-spoon recoveries.

Material description for soil samples must include:

- Classification
- Unified Soil Classification symbol
- Secondary components and estimated percentages
- Color
- Plasticity
- Consistency
- Density
- Moisture content
- Texture/fabric/bedding and orientation
- Grain angularity
- Depositional environment and formation
- Incidental odors
- Photoionization detector reading(s)
- Staining.

Material description for rock samples must include:

- Classification
- Lithologic characteristics
- Bedding/banding characteristics
- Color
- Hardness
- Degree of cementation
- Texture
- Structure and orientation

- Degree of weathering
- Solution or void conditions
- Primary and secondary permeability
- Sample recovery
- Incidental odors
- Photoionization detector reading(s)
- Staining.

3.4 WELL CONSTRUCTION AND INSTALLATION

After the hole is drilled and logged, backfill hole as required for proper screen placement. The integrity of the aquitard will be restored by placing a bentonite plug of an appropriate thickness, either to the top of the aquitard (normal well installation) or to within 0.3 ft of the top of the aquitard (DNAPL well). Aquifer fill will be clean filter pack.

Normal screen placement for the water table (surficial) aquifer will be within 2 ft of the screen extending above the static water level. The bottom of the screen will rest no more than 6 in. from the bottom of the hole or backfill material, whichever is applicable.

NOTE: The end cap in DNAPL wells will rest on the bottom of the bottom of the hole, or bentonite backfill if applicable (Section 3.2).

Screen placement for a confined aquifer well will normally be at the top of the confined aquifer.

Screen lengths will not normally exceed 10 ft. If it appears advantageous in a given situation (e.g., to screen an entire aquifer which is thicker than 10 ft), approval must be sought on a case-by-case basis from the appropriate regulatory agency. Otherwise, wells will be screened as follows:

Thickness of Aquifer	Action
<10 ft	Screen entire aquifer
>10 ft <30 ft	Screen top 10 ft consider vertically nested well cluster
>30 ft	Install vertically nested well cluster

The installation of monitoring wells in uncased or partially cased holes will begin within 12 hours of completion of drilling, or if the hole is to be logged, within 12 hours of well logging, and within 48 hours for holes fully cased with temporary drill casings. Once installation has begun, work will continue until the well has been grouted and the drill casing has been removed.

Well screens, casings, and fittings will conform to National Sanitation Foundation Standard 14 or ASTM equivalent for potable water usage. These materials will bear the appropriate rating logo. If the logos are not present, a written statement from the manufacturer/supplier stating that the materials contain the appropriate rating must be obtained. Material used will be new and essentially chemically inert to the site environment.

Well screen and casing should be inert with respect to the groundwater; therefore, the selection of screen and casing material will be based on select field tests of aquifer chemistry and potential contaminants. The screen will be capped without sediment trap or DNAPL sampling cup, and lowered into the hole. The well casing will be pre-cut to extend 2-2.5 ft above ground surface. Prior to placement of the last piece of well casing, a notch or other permanent reference point will be cut, filed, or scribed into the top edge of the casing.

Screen slot size will be appropriately sized to retain 90-100 percent of the filter pack material, the size of which will be determined by sieve analysis of formational material (Section 3.4).

The tops of all well casing will be capped with covers composed of materials compatible with the products used in the well installation. Caps may either be vented, or a telescopic fit, constructed to preclude binding to the well casing caused by tightness of fit, unclean surfaces, or weather conditions. In either case, it should be secure enough to preclude the introduction of foreign material into the well, yet allow pressure equalization between the well and the atmosphere.

Filter pack material will be placed, lightly tamped, and leveled. Filter pack will extend from the bottom of the hole to a height of 1-2 ft above the top of the screen. The filter pack will be capped with a minimum of 1 ft of fine (Ottawa) sand to prevent the bentonite seal from infiltrating the filter pack. If the bentonite seal is placed as a slurry, a minimum of 2 ft of fine sand will be required.

If the hole is less than 20-ft deep, the filter pack may be poured into the annulus directly. If the hole is deeper than 20 ft, the filter pack must be tremied into place.

Granular filter packs will be chemically and texturally clean, inert, and siliceous.

Filter pack grain size will be based on formation grain-size analysis. The D30 (70 percent retained) sieve size multiplied by a factor of not less than 3 nor greater than 6 will be used to determine the appropriate grain size.

Calculations regarding filter pack volumes will be entered into the Field Logbook along with any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Bentonite seals will be no less than 2-ft thick nor more than 5-ft thick as measured immediately after placement. The normal installation will include a 5-ft seal. Thinner seals may be used in special cases. The final depth to the top of the bentonite seal will be measured and recorded.

3.4.1 Grout

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, type II) (see previous table)
- 0.4-1 part (maximum) (2-5 percent) bentonite
- 8-gal (maximum) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement. Grout placement will be performed using a commercially available grout pump and a rigid, side discharge tremie pipe.

The following will be noted in the Field Logbook: (1) calculations of predicted grout volumes; (2) exact amounts of cement, bentonite, and water used in mixing grout; (3) actual volume of grout placed in the hole; and (4) any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Well protective casings will be installed around all monitoring wells on the following day as the initial grout placement around the well. Any annulus formed between the outside of the protective casing and the borehole will be filled to ground surface with cement.

The construction of each well will be depicted as built in a well construction diagram. The diagram will be attached to the boring log and will graphically denote:

- Screen location, length
- Joint location
- Granular filter pack
- Seal
- Grout
- Cave-in
- Centralizers
- Height of riser
- Protective casing detail.

3.5 MONITORING WELL COMPLETION

Assemble appropriate decontaminated lengths of pipe and screen. Make sure these are clean and free of grease, soil, and residue. Lower each section of pipe and screen into the borehole, one at a time, screwing each section securely into the section below it. No grease, lubricant, polytetrafluoroethelyne tape, or glue may be used in joining the pipe and screen sections.

If a well extends below 50 ft, centralizers will be installed at 50 ft and every 50 ft thereafter except within screened interval and bentonite seal. Centralizer material will be PVC, polytetrafluoroethelyne, or stainless steel. Determination of centralizer material will be based on the same criteria as screen and calling selection.

Cut the riser with a pipe cutter approximately 2-2.5 ft above grade. All pipe cuts MUST be square to ensure that the elevation between the highest and lowest point of the well casing is less than or equal to 0.02 ft Notch, file, or otherwise permanently scribe a permanent reference point on the top of the casing.

Torches and saws may not be used to cut the riser. Care must be taken that all filings or trimmings cut from the reference point fall outside the riser rather than into the well. **Under no circumstances will a permanent marker or paint pencil be used to mark the reference point**.

In some locations, safety requirements may mandate that a well be flush-mounted with no stick-up. If a flush-mounted well is required at a given location, an internal pressure cap must be used instead of a vented cap to ensure that rainwater cannot pool around the wellhead and enter the well through the cap.

When the well is set to the bottom of the hole, temporarily place a cap on top of the pipe to keep the well interior clean.

Place the appropriate filter pack (Section 3.4). Monitor the rise annulus with a weighted tape to assure that bridging is not occurring.

After the pack is in place, wait 3-5 minutes for the material to settle, tamp and level a capped PVC pipe, and check its depth weighted steel tape.

Add a 1-2 ft cap of fine-grained (Ottawa) sand to prevent infiltration of the filter pack by overlying bentonite seal. See Section 3.4 for guidance on appropriate thickness of fine sand layer.

Install the bentonite seal (2- to 5-ft thick) by dropping bentonite pellets into the hole gradually. If the well is deeper than 30 ft, a tremie pipe will be used to place either bentonite pellets or slurry. Tamp and level pellets. If the well is 30 ft, tamp with a capped PVC pipe, if >30 ft, tamping bay be accomplished with the weighted end of the tape. In either case, check the depth to the top of the seal with a weighted tape as above.

If the bentonite pellets are of poor quality, they may have a tendency to hydrate and swell inside the tremie pipe and bridge. This situation may be solved by the following procedure:

1. Use a different brand of pellets. Different brands may have longer hydration times.

2. Freeze the pellets[7]. Note that this will require a longer wait time to allow proper hydration after the pellets thaw.

3. Place the bentonite seal as a slurry using a side-discharge tremie pipe as though installing grout. Note (Section 3.4) this will require that a minimum of 2 ft of fine sand be placed as a cap on top of the filter pack material.

Wait for the pellets to hydrate and swell. Hydration times will be determined by field test or by manufacturer's instructions. Normally this will be 30-60 minutes. Document the hydration time in the field notebook. If the pellets are above the water level in the hole, add several buckets of clean water to the boring. Document the amount of water added to the hole.

Mix an appropriate cement-bentonite slurry (Section 3.4). Be sure the mixture is thoroughly mixed and as thick as is practicable.

Lower a side discharge tremie pipe into the annulus to the level of the pellet seal.

Pump the grout slurry into the annulus while withdrawing the tremie pipe and temporary casing.

Stop the grout fill at 5 ft below the ground surface. Allow to cure for not less than 12 hours. If grout settles more than 6 in., add grout to bring level back up to within 5 ft of ground surface. Place approximately 2 ft of bentonite pellets (minimum 0.5 ft) in annulus. Seat the protective casing in the bentonite seal, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Fill inner annulus (between well casing and protective casing) with bentonite pellets to the level of the ground surface. Cover bentonite pellets with 1 ft of clean granular material (coarse sand or pea gravel filter pack). Fill the outer annulus (between the protective casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing. Lock the cap.

— OR —

Continue the grout fill to the ground surface. Seat the protective casing in the grout, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Lock the cap.

— AND —

Allow the grout slurry to set overnight.

7. Bentonite pellets may be "flash-frozen" by brief immersion in liquid nitrogen (LN2). This can be accomplished by pouring LN2 over a small quantity (0.25-0.5 bucket) of pellets, allowing the LN2 to boil off, then pouring the pellets into the tremie pipe. **NOTE:** Use of LN2 is an additional jobsite hazard and must be addressed in the contractor's Health and Safety Plan. This contingency must be covered before drilling starts in order to avoid delays in well installation.

Fill the outer annulus (between the casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing.

Slope the ground surface away from the casing for a distance of 2 ft, at a rate of no less than 1 in. in 2 ft. Surface this sloping pad with a geotextile mat covered by 3 in. of coarse gravel.

— OR —

Frame and pour a 4-ft square × 6-in. thick (4 ft × 4 ft × 6 in.) concrete pad centered around the protective casing.

— AND —

Set pre-painted protective steel pickets (3 or 4) evenly around and 4 ft out from well. These pickets will be set into 2 ft deep holes, the holes will then be filled with concrete; and if the pickets are not capped, they will also be filled with concrete.

3.6 WELL DEVELOPMENT

Well development is the process by which drilling fluids, solids, and other mobile particulates within the vicinity of the newly installed monitoring well have been removed while restoring the aquifer hydraulic conductivity. Development corrects any damage to or clogging of the aquifer caused by drilling, increases the porosity of the aquifer in the vicinity of the well, and stabilizes the formation and filter pack sands around the well screen.

Well development will be initiated after 48 consecutive hours but no longer than 7 calendar days following grouting and/or placement of surface protection.

Two well development techniques, over pumping and surging, will be employed in tandem. Over pumping is simply pumping the well at a rate higher than recharge. Surging is the operation of a plunger up and down within the well casing similar to a piston in a cylinder.

3.6.1 Materials Required

The following materials will be required for well development:

- Well Development Form
- Boring Log and Well Completion Diagram for the well
- Submersible pump or bailer of appropriate capacity, and surge block
- Conductivity, pH, ORP, turbidity, dissolved oxygen, and temperature meters
- Electric well sounder and measuring tape
- Containers for purged water, if required.

3.6.2 Summary of Procedures and Data Requirements

Pump or bail the well to ensure that water flows into it, and to remove some of the fine materials from the well. Removal of a minimum of one equivalent volume is recommended at this point. The rate of removal should be high enough to stress the well by lowering the water level to approximately half its original level. If well recharge exceeds 15 gpm, the requirement to lower the head will be waived.

Slowly lower a close-fitting surge block into the well until it rests below the static water level, but above the screened interval. (NOTE: This latter is not required in the case of a light non-aqueous phase liquid well.)

Begin a gentle surging motion which will allow any material blocking the screen to break up, go into suspension, and move into the well. Continue surging for 5-10 minutes, remove surge block, and pump or bail the well, rapidly removing at least one equivalent volume.

Repeat previous step at successively lower levels within the well screen until the bottom of the well is reached. Note that development should always begin above, or at the top of, the screen and move progressively downward to prevent the surge block from becoming sand locked in the well casing. As development progresses, successive surging can be more vigorous and of longer duration as long as the amount of sediment in the screen is kept to a minimum.

Development is expected to take at least 2 hours in a small well installed in a clean sand, and may last several days in large wells, or in wells set in silts with low permeabilities.

Development will continue until little or no sediment can be pulled into the well, and target values for parameters listed below are met.

At a minimum, development will remove 3-5 well volumes of water. One development volume (DV) is defined as (1) equivalent volume, plus (1) the amount of fluid lost during drilling, plus (1) the volume of water used in filter pack placement.

1. Monitor water quality parameters before beginning development procedures, and after removing 2, 2.5, and 3 well volumes of water.

2. If these parameters have stabilized over the three readings, the well will be considered developed.

3. If the parameters have not stabilized after these three readings, continue pumping the well to develop, but stop surging. Monitor the stabilization parameters every half DV.

4. When the parameters have stabilized over three consecutive readings at half DV intervals, the well will be considered developed.

All water removed must be disposed of as directed by the Work Plan.

Record all data as required on a Well Development Record Form (Appendix A), which is made a part of the complete Well Record. These data include:

- Depths and dimensions of the well, casing, and screen obtained from the well diagram.
- Water losses and uses during drilling, obtained from the boring log for the well.
- Measurements of the following indicator parameters: turbidity, pH, conductivity, ORP potential, dissolved oxygen, and temperature.
- Target values for the indicator parameters listed above are as follows: pH – stabilize, conductivity – stabilize, ORP – stabilize, dissolved oxygen –- stabilize, temperature – stabilize, turbidity – 5 nephelometric turbidity units or stabilize. A value is considered to have stabilized when three consecutive readings taken at half DV intervals are within 10 percent of each other.
- Notes on characteristics of the development water.
- Data on the equipment and technique used for development.
- Estimated recharge rate and rate/quantity of water removal during development.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during well development activities.

6. REFERENCES

Aller, L. et al. 1989. Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells, National Water Well Association.

American Society for Testing and Materials (ASTM). D2487-92 Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System).

———. D5092-90 Standard Practice for Design and Installation of Groundwater Monitoring Wells in Aquifers.

Cohen, R.M. and J.W. Mercer. 1993. DNAPL Site Evaluation, CRC Press, Inc.

Nielsen, D.M. 1993. Correct Well Design Improves Monitoring, in *Environmental Protection*, Vol.4, No.7. July.

U.S. Army Toxic and Hazardous Materials Agency. 1987. Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition and Reports. March.

U.S. Environmental Protection Agency. 1989. Groundwater Handbook.

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Appendix A

Field Record of Well Development Form

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FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:
Well Volume Calculations	
A. Depth To Water (ft):	D. Well Volume/ft:
B. Total Well Depth (ft):	E. Total Well Volume (gal)[C*D]:
C. Water Column Height (ft):	F. Five Well Volumes (gal):

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

NOTE: NTU = Nephelometric turbidity unit.
ORP = Oxidation-reduction potential.

COMMENTS AND OBSERVATIONS: _____

FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Standard Operating Procedure No. 039
for
Sample Preservation and
Container Requirements

Prepared by

EA Engineering, Science, and Technology, Inc., PBC
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Hunt Valley, Maryland 21031

Revision 2
September 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 039	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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CONTENTS

DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
2	25 September 2018	Add notes about incremental sampling and minor changes	Daniel Hinckley, Sanita Corum	Matthew Bowman

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

2. MATERIALS

The following materials may be required:

- Containers (Section 3 provides a description)
- Nitric acid
- Sulfuric acid
- Sodium hydroxide
- Ice chests
- Ice.

3. DEFINITION OF CONTAINER TYPES

Listed below are the definitions of various container types.

Type	Container	Closure	Septum
A	80-ounce amber glass, ring handle bottle/jug, 38-millimeter (mm) neck finish	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm polytetrafluoroethelyne (PTFE) liner	
B	40-milliliter glass vial, 24-mm neck finish	White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size	24-mm disc of 0.005-inch) PTFE bonded to 0.120-inch silicon for total thickness of 0.125 inches
C	1-liter high density polyethylene, cylinder-round bottle, 28-mm neck finish	White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner	
D	120-milliliter wide mouth glass vial, 48-mm neck finish	White polyethylene cap, 40-480 size; 0.015-mm PTFE liner	
E	250-milliliter Boston round glass bottle	White polypropylene or black phenolic, open top, screw cap	Disc of 0.005-inch PTFE bonded to 0.120-inch silicon for total thickness of 0.125 inches
F	8-ounce short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner	
G	4-ounce tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner	

Type	Container	Closure	Septum
H	1-liter amber, Boston round, glass bottle, 33-mm pour-out neck finish	White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner	
K	4-liter amber glass ring handle bottle/jug, 38-mm neck finish.	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner	
L	500-milliliter high-density polyethylene, cylinder bottle, 28-mm neck finish	White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner	

4. PROCEDURE

All containers described in Section 3 must be certified clean (SOP Number [No.] 031), with copies of laboratory certification furnished upon request. There may be circumstances when alternative containers will be used (e.g., aluminum foil around tissue samples placed in plastic bags, plastic buckets or bags for large soil/sediment samples, etc.) for which laboratory certification may not be available. Such containering should be appropriately decontaminated or verified appropriately clean prior to using.

Water samples will be collected into pre-preserved containers appropriate to the intended analyte as documented in the Quality Assurance Project Plan. Samples taken for volatile organic compounds will be collected in accordance with SOP No. 003, Section 3.3.8. Samples taken for metals analysis will be verified in the field to a pH <2. The container should be tightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure that this operation may have generated. Samples taken for total phosphorous content will be verified in the field to a pH <2. The container should be tightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure that this operation may have generated. Samples taken for cyanide will be verified for a pH >12. Most other samples do not require added preservation; however, there are analytes that may require special preservation, (i.e., sulfide that requires a zinc acetate preservation). Preservation must be performed as documented in the project-specific Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2 degrees Celsius (°C).

Soil and sediment samples will be collected into containers appropriate to the intended analyte as documented in the Quality Assurance Project Plan. Samples taken for volatile organic compound analysis will collected in accordance with the site-specific SOP. Samples taken for metals analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Large (1-2 kilograms) soil/sediment samples taken for incremental samples (SOP No. 057) can be placed in pre-cleaned (SOP No. 005) gallon plastic bags or plastic buckets. Under most circumstances, no preservatives will be added to soil or sediment samples; follow project-specific requirements as documented in the Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

5. MAINTENANCE

Not applicable.

6. PRECAUTIONS

Note that acidifying a sample containing cyanide may liberate hydrogen cyanide gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces (i.e., a vehicle).
- Hold suspected hydrogen cyanide-generating sample away from body and downwind while manipulating it.
- See the Health and Safety Plan for other safety measures.

7. REFERENCES

U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste, SW-846.

———. 1987. A Compendium of Superfund Field Operations Methods, EPA 540-P87-001.

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Standard Operating Procedure No. 042
for
Disposal of
Investigation-Derived Material

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Revision 1
December 2014

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CONTENTS

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to define the required steps for disposing of investigation-derived material (IDM) generated during field activities.

IDM, as used herein, includes soil cuttings, drilling muds, extraneous sediment, purged groundwater, decontamination fluids, and disposable personal protective equipment. For the sake of clarity and ease in use, this Standard Operating Procedure is subdivided into procedures for disposal of liquid IDM and solid IDM as follows:

- Liquid IDM (Section 3.2) includes the following materials:
  - Water from initial development of new wells and the redevelopment of existing wells
  - Purge water from groundwater sampling
  - Decontamination fluids (Section 3.4)
- Solid IDM (Section 3.3) consists of the following materials:
  - Soil drill cuttings from monitoring well installation
  - Sediment remaining after collection of the required sample volume
  - Grout, a mixture of cement and bentonite, generated during installation of monitoring wells
  - Disposable personal protective equipment (Section 3.4).

2. MATERIALS

The following materials may be required:

Any additional equipment that may be dictated by project or site-specific plans	Hazardous waste labels
Bar codes	Permanent marker
Chain-of-custody forms	Field logbook (bound)
Department of Transportation 17C specification metal containers	Waste identification labels

3. PROCEDURE

3.1 GENERAL

No container will be labeled as a “Hazardous Waste” unless the contents are in fact known to be hazardous as defined by 40 Code of Federal Regulation 261.

IDM may be disposed onsite if it is: (1) initially screened, or evaluated to determine whether it is contaminated; (2) not abandoned in an environmentally unsound manner; and (3) not inherently waste-like.

IDM is to be considered contaminated if: (1) it is visually or grossly contaminated; (2) it has activated any field monitoring device that indicates that the level exceeds standard Level 1; (3) it has previously been found to exhibit levels of contamination above environmental quality standards; and (4) the responsible party and/or appropriate regulator deem(s) that records of historical uses indicate that additional testing of the IDM is needed, or additional caution is warranted handling IDM from a given site.

3.2 PROCEDURES FOR LIQUID INVESTIGATION-DERIVED MATERIAL DISPOSAL

Listed below are the procedures for the disposal of liquid IDM:

1. All water from the initial development of new wells, and purge water generated during the first round of groundwater sampling, will be containerized in Department of Transportation approved 55-gallon drums. Decontamination fluids may be bulk-containerized until completion of the field task.

2. Label all containers as to type of media, date the container was sealed, point-of-generation, and points-of-contact. The well number and container number will be identified on the container.

3. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include the following: date of generation, contents of containers, number of containers with the same contents (if applicable), location of containers, well number the media is associated with, personnel sampling the media, sampling dates, and sampling results.

4. Containers of well development water and purge water may be stored at the well site pending the first round analytical results.

5. Laboratory turnaround time must be no greater than 30 days. Upon receipt of the analytical results, a copy will be furnished to the client within 3 working days. Both the client and contractor will evaluate the data to determine disposal requirements, per state and local regulations. A disposal decision is required within 10 days of receipt of sampling results. Appropriate disposal must be performed no later than 50 days from the

1. This value is defined as two times background, where "background" values are to be determined as follows: (1) regional background values will be used where they are available; and (2) if regional values are not available, background may be empirically determined at uncontaminated sampling sites using onsite sensors such as organic vapor analyzers (photoionization detector or flame ionization detector), scintillometers, etc.

decision date unless prevented by inclement weather (e.g., rain and muddy conditions may preclude site access, freezing weather may freeze media).

Dispose of media in accordance with Steps 6 and 7 of this procedure, as appropriate.

6. If the first round analytical data of the liquid media are below the Maximum Contaminant Levels established by the Federal Safe Drinking Water Act, the water may be gradually infiltrated into the ground at least 50 feet downgradient of the well.

   If the well location has no downgradient area, the water will be infiltrated into the ground in an area deemed appropriate by the client and the contractor/support personnel.

   Disposal locations must allow percolation of the water and prohibit "ponding."

   Upon completion of water discharge to ground, enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook and provide this information to the client.

   Empty containers are to be properly decontaminated, stored, and reused by the appropriate personnel.

   If the liquid media sampling results do not meet the required Maximum Contaminant Levels and cannot be discharged to the ground, then determine if the waste meets the sanitary sewer discharge criteria (National Pollutant Discharge Elimination System standards).

7. If, at any time, visual contamination of purge/development water is observed, or if organic vapor monitor readings (HNu, photoionization detector) register more than 5 parts per million above background and/or radiological meters register more than twice the background mrems, then the liquid will be drummed and a composite sample will be taken that day. A disposal decision will be based on the analytical results of this sample rather than the first round of analytical results.

3.3 PROCEDURES FOR SOLID INVESTIGATION-DERIVED MATERIAL DISPOSAL

Listed below are the procedures for the disposal of solid IDM:

1. If the conditions outlined in Section 3.1 are met, proceed to Section 3.3, Step 2; otherwise, proceed to Section 3.3, Step 7.

2. During soil drilling operations or sediment sampling, the resulting cuttings, mud, and/or extraneous sediment will be discharged onto the ground (or waterbody for sediment) near the well (or sample location for sediment) if the following conditions are met: (1) no visual contamination is observed, (2) organic vapors are less than 5 parts per million

above background, (3) radiological meter readings (if applicable) are under two times background, and (4) the medium has been screened and found to be less than two times background if the potential for contamination exists.

Proper sediment and erosion control measures will be implemented as follows:

- Soil drill cuttings will be uniformly spread and contoured to blend with the surroundings of the site.
- If amount of solid IDM exceeds 5,000 square feet or 100 cubic yards of material, a sediment and erosion control plan is required.
- If the amount of solid IDM is under 5,000 square feet or 100 cubic yards, the site will be stabilized as soon as possible. Stabilization includes mulch, seed, and tack.
- Critical areas require stabilization within 7 days from the date of well completion. Critical areas include swales, water sources, drainage ditches, etc.
- All other disturbed areas require stabilization within 14 days from the date of well completion.

3. If the well location is in or near a wetland, the soil drill cuttings will be drummed and transported away from the site for spreading.

4. Label all IDM containers that will not be spread on the day of generation. Each container should be labeled with the type of media, date the container was sealed, point-of-generation, and name of the contact person. The well number or sample location and container number should be identified on the container.

5. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include: date of generation, contents in containers, number of containers with the same contents, location of containers, and well number or sample location the media is associated with.

6. Containers will be staged at the well site until contractor/support personnel spread the cuttings in the appropriate locations, using proper sediment and erosion control measures per Section 3.3.

7. If soil drilling mud, cuttings, or sediment show visible contamination, or organic vapor readings are more than 5 parts per million above background levels, or radiological meter readings (if applicable) show greater than two times background levels, or if the potential for contamination exists (levels greater than two times background), media will immediately be containerized, labeled appropriately (Section 3.2), and sampled on the same day.

8. The solid IDM should be sampled and appropriate Toxicity Characteristic Leaching Procedure analyses conducted prior to determining disposition. Laboratory turn-around time must be no greater than 30 days. Upon receipt of analytical results, a copy will be furnished to the client within 3 working days. The contractor will evaluate the data to determine disposal requirements within 10 days. Appropriate disposal must be performed no later than 50 days after the decision date if weather permits (Section 3.2).

    - If the solid IDM is determined to be non-hazardous and uncontaminated, proceed to Section 3.3.

    - If the solid IDM is determined to be non-hazardous but contaminated, proceed to Section 3.3.

    - If the solid IDM is found to be hazardous wastes, proceed to Section 3.3.

9. If the solid IDM is not a hazardous waste **and** analytical data show contaminant concentrations below the U.S. Environmental Protection Agency Region 3 (or applicable Region where work is being performed) Risk-Based Concentrations, contact the appropriate federal, state, or local agency for approval to discharge onto the ground or back to the waterbody near the site of generation.

    - Follow steps detailed in Section 3.3, Step 2 (above) pertaining to sediment and erosion control.

    - Upon completion of the solid IDM discharge to the ground or waterbody (for sediment), enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook. This information must be provided to the client.

    - Empty containers are to be properly decontaminated, stored, and reused by appropriate personnel.

10. If the intrusive media is not a hazardous waste but analytical data show concentrations above the screening criteria, dispose of the IDM according to state and local regulations.

    - Ensure that the waste containers are properly labeled as applicable in accordance with Section 3.3, Step 4.

    - Inform the client of the type and amount of waste, and location of the waste.

    - When the waste is removed, enter the type of waste, amount of waste, date of pickup, and destination of the waste in a bound Field Logbook. This information must be provided to the client.

3.4 PROCEDURES FOR DECONTAMINATION SOLUTION AND PERSONAL PROTECTIVE EQUIPMENT DISPOSAL

Decontamination solutions include catch water from steam-cleaning operations performed on large sampling equipment, drill rigs, and drums, as well as smaller quantities of soapy water and rinse solutions used in decontaminating field sampling equipment. At the completion of the field event, a composite sample of the decontamination solution will be taken. The decontamination solution will be treated as liquid IDM pending results (Section 3.2).

Personal protective equipment will be containerized onsite, appropriately labeled, and disposed in a designated trash receptacle.

4. MAINTENANCE

The waste manifest document and bill of lading should be uploaded to the project file as soon as possible in either hard copy or electronic format. Refer to EA's Records Retention Policy for archiving information.

5. REFERENCES

Environment Article Section 7-201(t).

U.S. Environmental Protection Agency. 1991. Management of Investigation-Derived Wastes during Site Inspections PB91-921331, OERR Directive 9345.3-02. Office of Emergency and Remedial Response U.S. Environmental Protection Agency, Washington, D.C. May.

Standard Operating Procedure No. 046
for
Aqueous Diffusion Samplers

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1. SCOPE OF APPLICATION

The purpose of this Standard Operating Procedure is to establish the protocol for collecting groundwater samples using aqueous diffusion samplers. The procedure is designed to permit the collection of representative groundwater samples for analysis of volatile organic compounds (VOCs).

2. CONSTRUCTION OF AQUEOUS DIFFUSION SAMPLERS

The aqueous diffusion samplers are constructed by sealing de-ionized water in a 2-in. diameter × 1-mil thick polyethylene tubing. The de-ionized water is sealed in the poly tubing by using a heat seal device. One end of the poly tube is rolled over onto itself several times, then heat is applied to seal this end. The poly tube is then filled with de-ionized water. The top end (unsealed end) of the tube is then rolled over onto itself until there is no headspace in the poly tube; heat is then applied to seal this end. Care is taken to ensure that no headspace or air bubbles are present in the tube prior to sealing the top end. Each diffusion sampler is approximately 2 ft in length. The samplers are weighted with stainless steel bolts enclosed in 4-mil polyethylene tubing attached to the bottom of the sampler, and a string is attached to the top of the sampler for placement and retrieval.

3. EQUIPMENT/MATERIALS

3.1 AQUEOUS DIFFUSION SAMPLER PLACEMENT

- Well construction data, location map, and field data from the previous sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling form (Figure SOP046-1).
- Electronic water level measuring device, 0.01-ft accuracy for monitoring water level prior to installation of the diffusion sampler.
- Diffusion sampler constructed of 2 ft length × 2 in. width 1-mil polyethylene lay-flat tubing filled with de-ionized water and weight attached to bottom.
- Twine, string, or rope. The depth of each sampler should be established prior to field placement so enough twine, string, or rope is available for installation.

3.2 AQUEOUS DIFFUSION SAMPLER RETRIEVAL

- Volatile organic analyte sample bottles and sample preservation supplies (as required by the analytical methods) needed for diffusion sampler retrieval.

- Sample tags or labels.

- Cooler with bagged ice for storage of sample bottles during shipment to a laboratory.

3.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing aqueous diffusion sampler installation and retrieval. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and installation and retrieval form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, and record pertinent observations in field logbook and field sampling form.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with a photoionization detector instrument at the rim of the well and in the breathing zone, and record the readings in the field logbook and the field sampling form.

- Measure and record the height of protective casing above the concrete pad or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before installation of the aqueous diffusion sampler begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will be measured prior to installation of the sampler.

3.4 SAMPLING PROCEDURE

The following general procedures should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and field sampling form, as appropriate, prior to installation of the diffusion sampler: date and time of sampler installation, depth of sampler, and total depth of well.

- Prepare the diffusion sampler by attaching weight at the base of the sampler and a string to the top of the sampler.

- Install the sampler at the predetermined depth. Depth of sampler will be determined on a well-by-well basis based on previous low-flow sampling data, or previously collected aqueous diffusion samplers.

- Allow the diffusion to equilibrate for approximately 21 days. Return after approximately 21 days to retrieve the sampler.

- Enter the following information in the field logbook and field sampling form, as appropriate, during retrieval of diffusion sampler: date and time of sampler retrieval, analytical method, and quality assurance/quality control as necessary.

- Retrieve the diffusion sampler from the well.

- After retrieval is complete, remove string and weight, and make a diagonal cut towards the top of the sampler. The diagonal cut allows easier filling of the sample containers.

- Begin filling the sample containers from the diagonal cut, allowing the water to fill the volatile organic analyte sample containers by allowing the water to flow gently down the inside of the container with as little agitation or minimal aeration as possible.

- Label each sample as it is collected. Samples will be placed into a cooler with ice for delivery to a laboratory.

- After collection of the samples, the wells will be capped and locked.

- Complete remaining portions of field sampling form after each well is sampled, including sample date and time (time of retrieval from the well), well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of the sampling event.

3.5 SAMPLE PRESERVATION

- **VOCs**—Fill the sample bottle pre-preserved with hydrochloric acid, seal with a teflon-lined cap, and place in a cooler with ice for shipment to a laboratory. Cooler will maintain a temperature of 4°C for shipment to the laboratory.

Note that aqueous diffusion samplers are not submitted for other laboratory analytical parameters.

3.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. Field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each sample delivery group (a sample delivery group may not exceed 20 samples) at the frequency noted:

- ***Equipment Blank***—One aqueous diffusion sampler will be constructed and submersed in a sealed container with de-ionized water for the 21-day equilibration period. This equipment duplicate should be analyzed the same as the other samplers to determine if materials used for aqueous diffusion samplers may have outgased VOCs, or otherwise affected laboratory analytical results.

- ***Field Duplicate***—Required at a frequency of 10 percent per sample delivery group, as per the site Long-Term Monitoring Plan.

- ***Matrix Spike/Matrix Spike Duplicate***—Required at a frequency of 5 percent, as per the site Long-Term Monitoring Plan.

- ***Trip Blank***—Required for VOC samples at a frequency of one per sample shipment, as per the site Long-Term Monitoring Plan.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:		Project Number:	
Well ID:		Well Lock Status:	
Well Condition:		Weather:	

Gauge Date:		Gauge Time:	
Sounding Method:		Measurement Ref:	
Stick Up/Down (ft):		Well Diameter (in.):	

Purge Date:		Purge Time:	
Purge Method:		Field Personnel:	
Ambient Air VOCs (ppm):		Well Mouth VOCs (ppm):	

WELL VOLUME

A. Well Depth (ft):		D. Well Volume/ft (L):	
B. Depth to Water (ft):		E. Well Volume (L) (C*D):	
C. Liquid Depth (ft) (A-B)		F. Three Well Volumes (L) (E*3):	

G. Measurable LNAPL? Yes _____ /ft No _____

Parameter	Beginning	1	2	3	4	5
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Total Quantity of Water Removed (L):			
Samplers:		Sampling Time (Start/End):	
Sampling Date:		Decontamination Fluids Used:	
Sample Type:		Sample Preservatives:	
Sample Bottle IDs:			
Sample Parameters:			

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Standard Operating Procedure No. 047
Direct-Push Technology Sampling

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1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes the protocol for using direct-push technology (DPT) in media sampling and performing subsurface characterization. This SOP includes the following DPT methods: Geoprobe®, Hydropunch®, Cone Penetrometer Testing (CPT), and Site Characterization and Analysis Penetrometer System (SCAPS).

2. MATERIALS

The following materials may be required:

Appropriately sized, all-terrain vehicle-skid-or track-mounted; DPT equipment; and supplies (i.e., hydraulic derrick and hammer assembly)	Personal protective equipment
Bentonite grout and clean sand for DPT hole abandonment	Phosphate-free, laboratory-grade detergent (e.g., Liquinox, Alconox, etc.)
DPT stainless steel rods	Source of approved water
Heavy plastic sheeting	Steam cleaner/sprayer and water obtained from approved source for decontaminating DPT equipment
Logbook	Steel drums for intrusion derived wastes (e.g., contaminated personal protective equipment, decon solutions, etc.)
Long-handled bristle brushes	Wash and rinse tubs
Mini-bailer or tubing and peristaltic pump (groundwater sampling only)	

3. GEOPROBE® AND HYDROPUNCH®

3.1 MATERIALS

Water sources for Geoprobe® and Hydropunch® activities, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the Geoprobe® and Hydropunch® equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the Geoprobe® and Hydropunch® equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

3.2 GROUNDWATER – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site to facilitate aquifer characterization and analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all sampling and installation procedures, and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® and Hydropunch® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means.

- Drive the sampler to the desired groundwater sampling interval. At the desired depth, insert extension rods down the inside diameter of the probe rods until the extension reaches the bottom of the screen. Remove the probe rods and sampler sheath while holding the screen in place.

- Collect the groundwater sample in the screen interval with a mini-bailer, peristaltic or vacuum pump, or other acceptable small diameter sampling device.

- The head of the rod may be equipped with a sensing device for characterization of soil properties or the contaminant content.

The subcontractor will use the equipment-specific guidelines for installation of the Hydropunch® equipment. Rods will be forced into the ground by hydraulic means.

- The Hydropunch® tool is a double cylinder, designed to be sealed until the desired sampling depth is reached. Upon reaching the desired sampling depth, the outer cylinder is pulled back, exposing a perforated, stainless steel sampling barrel covered with filter material.

- The water sample enters the barrel and the sample is retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly. Groundwater is the only media that is sampled by Hydropunch® equipment.

- The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

- The sample volume collected with this technique is approximately 500-1,000 ml. Larger sample volumes can be collected by inserting tubing attached to a peristaltic pump into the rods to obtain water samples.

If desired, a small diameter monitoring well may be installed at this point. Refer to SOP No. 019 (Monitoring Well Installation).

If a well will not be installed, the rods will be removed as the borehole is simultaneously filled with a bentonite/grout mixture. A polyvinyl chloride (PVC) tube fed into the rod casing will allow the addition of grout.

3.3 SUBSURFACE SOIL – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site for characterization of the stratigraphy and for analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all DPT sampling and soil characterization. All procedures and soil characteristics will be fully documented in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means. Additional rods will be added in 3- to 4-ft increments until the leading edge of the sampler reaches the top of the desired sampling interval.

Once the desired sampling depth has been reached, insert extension rods down the inside diameter of the probe rods until it reaches the top of the sampler assembly. Attach the extension rod handle to the top extension rod. Turn the handle clockwise until the stop-pin detaches from the drive head. Remove the extension rods and the stop-pin. Attach a drive cap to the probe and drive the sampler approximately 2 ft using hydraulic derrick.

The DPT sampler can be retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly.

The liner will be capped with Teflon® tape and vinyl end caps. The liners can be split open to remove samples for composition analysis or for transfer to other containers for shipment to the laboratory for analysis.

The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

3.4 DECONTAMINATION

All Geoprobe® and Hydropunch® DPT equipment must be thoroughly cleaned before and after each use to allow retrieval of representative groundwater samples. Geoprobe® soil sample liners are disposed of after each use. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

After decontamination, a new clean liner will be installed and all parts will be inspected for wear or damage.

Refer to SOP No. 005 (Field Decontamination).

3.5 ABANDONMENT

Pure bentonite or a bentonite/grout mixture (20:1) will be used to fill the resulting borehole if the water table is penetrated. Boreholes that do not penetrate the water table will be backfilled with cuttings from the hole and topped with a bentonite seal. Clean sand will be used to fill any remaining volume in the borehole.

Abandonment of Geoprobe® and Hydropunch® generated DPT boreholes will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

4. CONE PENETROMETER TESTING

4.1 MATERIALS

A CPT rig typically consists of an enclosed 20- to 40-ton truck equipped with vertical hydraulic rams that are used to force a sensor probe into the ground. The weight of the CPT rig is dependent upon the thrust required at the site. The majority of CPT rigs are mounted in heavy-duty trucks that are ballasted to a total dead weight of approximately 15 tons. Screw anchors are utilized to develop the extra reaction to reach the maximum thrust of 20 tons. The rig is separated into two separate workspaces: data acquisition and hydraulic push areas.

Water sources for CPT activities and decontamination must be approved by the Project Manager prior to arrival of the CPT equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the DPT equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

4.2 SUBSURFACE CHARACTERIZATION

The objective of this technology is to collect stratigraphic information using CPT equipment to determine subsurface stratigraphy and geotechnical properties at a particular site. CPT activities will be in accordance with American Society for Testing and Materials D 3441-86 and American Society for Testing and Materials D 5778-95. The stratigraphic information gathered can be used to facilitate the selection of DPT sampling screen intervals. At the same time, it is possible to install a 0.25-in. diameter pre-packed PVC monitoring well.

CPT rods are used to hydraulically push the CPT probe into the subsurface. Probes cannot be pushed into hard rock, and significant gravel or cobble content in the formation may impede or preclude penetration of the probe. The depth of penetration achievable depends on the type of formation, type of sampling probe, and size of the hydraulic equipment used.

The CPT probe includes the following components:

- A conical tip to measure vertical resistance beneath the tip.
- A friction sleeve to measure frictional resistance on the side of the probe, as a function of depth.

- Two internal strain gauge-type load cells, which independently measure the vertical resistance and side friction.

- A cone pressure gauge to measure the water pressure as the probe is pushed into the ground.

- Inclinometer to determine potential drifting of the probe (optional).

- Seismic transducers to perform downhole seismic surveys (optional). Therefore, stratigraphic data collected with the CPT include: tip resistance, local friction, friction ratio, pore pressure, and resistivity.

Data will be transferred from the probe to the data acquisition system or logger through an electrical cable. The hole will be advanced continuously at a rate of 0.6-1.0 in. per second. The data will be logged at every 0.4-0.8 in. of penetration. Monitor the probe's stratigraphic position will be monitored as it advances downward. Perform pore water pressure dissipation tests in representative hydrostratigraphic intervals. Record dissipated pore water pressures to represent hydraulic head values.

Once the confining unit underlying the surficial aquifer or the required depth has been reached, the CPT is pulled from the ground. Target interval samples can be collected during CPT hole advancement using direct push sampling techniques, i.e., Geoprobe® or Hydropunch® (Section 3).

4.3 DECONTAMINATION

All CPT equipment must be thoroughly cleaned before arrival at the work site, between test holes, and prior to being moved out of a work area. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

Refer to SOP No. 005 (Decontamination).

4 .4 ABANDONMENT

If the push hole was developed for the stratigraphic test only, once the testing is completed, grout the hole from bottom to top. If the hole has not collapsed after removing the CPT, PVC piping will be used to grout the hole. If the hole has collapsed after removing the CPT, then hollow CPT rods and a sacrificial tip will be used to grout the hole. The PVC pipe or CPT rods will be pushed to the bottom of the hole. Grout will then be pumped to the bottom of the hole as the PVC pipe or CPT rods are withdrawn.

Refer to SOP No. 028 (Well and Boring Abandonment).

5. SITE CHARACTERIZATION AND ANALYSIS PENETROMETER SYSTEM

5.1 MATERIALS

SCAPS cone penetrometer and laser induced fluorescence (LIF) technology requires the use of a specialized 20-ton truck. The truck has two separate enclosed compartments. Each compartment is temperature controlled and monitored for air quality. The two rooms are the data acquisition and processing room, and the hydraulic ram/rod handling room. Approximately 20 ft of overhead clearance is required to fully extend the hydraulic ram and allow for leveling jack movement.

All materials required to complete SCAPS analysis are provided by the subcontractor to include cone penetrometer equipment. All hydraulic equipment, SCAPS rods, nitrogen lasers, etc. are included within the vehicle. A decontamination water source and a source of water for mixing the grout are required.

Water sources for equipment decontamination must be approved by the Project Manager prior to arrival of the SCAPS equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the SCAPS equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

5.2 HYDRAULIC PUSHING AND SAMPLING

The objective of the SCAPS technique is to allow grab samples and stratigraphic information to be collected at a selected site to facilitate subsurface characterization and for analysis of potential contaminants. The analytical results obtained can also be used to determine the placement of monitoring wells. At the same time, it is possible to install a small diameter well for sampling purposes. Refer to SOP No. 019 (Monitoring Well Installation). If a well will not be installed, the borehole can be grouted as the equipment is removed.

A site geologist will be present during all installation and sampling procedures and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole SCAPS equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after direct-push activities.

The subcontractor will use the equipment specific guidelines for installation of the SCAPS DPT equipment. Prior to SCAPS field activities, calibration soil samples will be collected and analyzed in order to determine the LIF sensor fluorescence threshold and detection limits for the site.

SCAPS LIF technology uses a pulsed nitrogen laser coupled with an optical detector to make fluorescence measurements via optical fibers. The LIF sensor is mounted on a cone penetrometer probe so that soil classification data and fluorescence data are collected simultaneously. The laser consumes nitrogen gas, which is supplied from cylinders stored on the accompanying trailer.

The SCAPS CPT sensors are used to gather stratigraphic information. See Section 4 for CPT operating procedures.

Target interval samples can be collected during SCAPS hole advancement using direct push sampling techniques such as Geoprobe® or Hydropunch® (Section 3).

5.3 DECONTAMINATION

Decontamination of SCAPS equipment is automated after initialization by a field team member. A pressurized hot water system is used to decontaminate the push rods as they are retracted from the ground. The SCAPS vehicle is equipped with a decontamination collar mounted to the bottom that cleans the rods. The decontamination water is removed by vacuum and transferred to a storage drum prior to disposal or treatment. A trailer attached to the back of the vehicle contains the water pump, heater for decontamination, and decontamination water containment drum.

Worker exposure is reduced by minimizing contact with contaminated media.

Refer to SOP No. 005 (Decontamination).

5.4 ABANDONMENT

SCAPS automatically grouts the penetrometer cavity as the rods are removed. The grout is pumped at high pressure through a 0.25-in. diameter tube in the center of the penetrometer rods. The tip is sacrificed at the bottom of the cavity to allow release of the grout.

A trailer attached to the back of the vehicle contains the 300-gal grout mixing bin and pump.

If the automatic grout feed does not work, the cavity will be manually filled with grout.

Abandonment of SCAPS generated borehole will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

6. MAINTENANCE

Not applicable.

7. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during intrusive activities.

8. REFERENCES

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Standard Operating Procedure No. 048
for
Low-Flow Sampling

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1. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING DEDICATED PUMPS

1.1 SCOPE OF APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for collecting groundwater samples using dedicated pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity.

1.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, and field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Electric water level measuring device, 0.01 ft accuracy for monitoring water level during pumping operations.
- Pumps: adjustable rate, submersible pumps constructed of stainless steel and Teflon®.
- Tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene tubing will be used.
- Flow measurement supplies (e.g., graduated cylinder and stop watch).
- Power source (generator, etc.).
- Water quality indicator parameter monitoring instruments—pH, turbidity, specific conductance, and temperature. Optional indicators—Eh and dissolved oxygen.
- Flow-through cell (preferred) or clean container for water quality probes.
- Decontamination supplies (for monitoring instrumentation).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.
- Drum for purge water containment.

1.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.

- Lay out sheet of polyethylene for monitoring and sampling equipment.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with an ionization detector (flame or photo) instrument at the rim of the well and in the breathing zone, and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Dedicated sampling pumps should be positioned with the pump intake mid-point in the screened interval. If non-dedicated equipment is used, care will be taken to position pump or sampling hose intake at the screen mid-point.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Prepare the pump by checking electrical connections, discharge tubing, and motor (Grundfos Redi-Flo2). Locate the generator (if applicable) downwind of the well; connect the power converter to the generator and to the pump.

1.4 WELL PURGING AND SAMPLING PROCEDURE

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Connect the flow-through cell or clean container containing the instrumentation header to the pump discharge and begin purging the well at 0.2-0.5 L/min, unless a different purge rate has been previously established for that well. Fill the flow cell completely. Care should be taken not to cause entrapment of air in the system. Record the purge start time and purge rate.

- Establish that the water level has not dropped significantly such that the pump is dry (bubbles in discharge) or water is heard cascading down the inside of the well. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.1-0.2 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collect the appropriate volume of samples with the pump.

- During purging of the well, monitor the water quality indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Record purge rate, volume purged, depth to water, water quality indicator parameters values, and clock time at 3- to 5-minute intervals in field logbook and sampling record. Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 nephelometric turbidity units (NTU) are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing. Record pertinent observations in field logbook and sampling records.

- Begin filling sample containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation are discussed in Section 1.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated pumping assembly to the well by hanging the tube, electric line, and support cable inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

1.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

— Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

— Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

— Invert the bottle, tap lightly, and check for air bubbles.

— If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

— Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

1.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each sample delivery group (SDG) (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.

- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.

- Equipment Rinsate Blank—Required once prior to installation of dedicated pump systems.

- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.

- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

1.7 DECONTAMINATION

Non-dedicated sampling equipment and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternative procedures must be approved by the Project Manager prior to sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

2. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING PERISTALTIC PUMPS

2.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting groundwater samples using peristaltic pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity, and is intended to be used in conjunction with the analyses for the most common types of groundwater contaminants (VOCs and inorganic compounds).

2.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Water level measuring device, 0.01 ft accuracy (electronic preferred) for monitoring water level drawdown during pumping operations.
- Peristaltic pump.

- In-well tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene, PVC, Tygon, or polyethylene tubing may be used.
- Pump head tubing: Silicon tubing must be used to in the pump head assembly.
- Flow measurement supplies (e.g., graduated cylinder and stop watch).
- Power source (battery, etc.).
- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.
- Flow-through cell (preferred) or clean container for water quality probe.
- Decontamination supplies (for monitoring instrumentation).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.
- Drum for purge water containment.

2.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.
- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.
- Unlock well and remove well cap (if applicable).
- Measure VOCs with an ionization detector (photo or flame) instrument at the rim of the well and in the breathing zone and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad, or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will not be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Position the intake of the sampling hose at the mid-point of the screened interval.

- Prepare the pump by checking electrical connections and discharge tubing. Locate the battery downwind of the well; connect the peristaltic pump to the battery.

2.4 WELL PURGING AND SAMPLING PROCEDURES

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Measure the water level with the pump in well before starting the pump. Begin purging the well at 0.3-0.5 L/min, unless a different purge rate has been previously established for that well.

- If well diameter permits, establish that the water level has not dropped significantly such that the pump is dry (air in discharge) or tubing suction is broken. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Care should be taken not to cause pump suction to be broken, or entrainment of air in the pump system. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.3 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collected the appropriate volume of samples with the pump.

- During purging of the well, monitor the field indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 NTU are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the sample discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing.

- Collect groundwater samples directly from the silicon tubing into preserved (when appropriate) sample containers. Begin filling sample containers from the pump discharge, allowing the water to fill the containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Detail on sample preservation are discussed in Section 2.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated tubing assembly to the well by hanging the tube inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including: sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

- The silicon tubing used in the peristaltic pump will be changed after use at each well.

2.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HCL, and check the pH. Adjust the volume of HCL to assure pH<2.

  — Add the amount of HCL determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down, and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

2.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent
- Equipment (Rinsate) Blank—Required once prior to installation of dedicated sample tubing
- Source Water Blank—Required at a frequency of one per source per sampling event
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.
- Temperature Blank—Required at a frequency of once per sample shipment container.

2.7 DECONTAMINATION

Non-dedicated sampling and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternate procedures must be approved by the Project Manager prior to the sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

3. SURFACE WATER AND LEACHATE SEEP SAMPLING PROCEDURE

3.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting surface water and leachate seep samples. The procedure is designed to permit the collection of representative surface water and leachate seep samples, and has been adapted from the procedure outlined in the Work Plan. This SOP is suitable for collecting surface water and seep samples requiring analyses for the most common types of surface water contaminants (VOCs and inorganic compounds).

3.2 EQUIPMENT/MATERIALS

- Work Plan.
- Location map, field data from last sampling event.
- Field logbook and Field Record of Surface Water and Sediment Sampling forms (Figure SOP048-2).
- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.
- Decontamination supplies (for monitoring instrumentation).
- Dedicated, pre-cleaned 1-L wide-mouth or volatile organic analyte sample container (for sample collection).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.

3.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing surface water or leachate seep sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, sample station identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Visually inspect sample station for evidence of changes in physical condition; record pertinent observations in field logbook and sampling form.

- Measure VOCs with a flame ionization detector instrument in the breathing zone and record the reading in the field logbook and sampling form.

3.4 SAMPLING PROCEDURE

The technique for surface water and leachate seep sampling must be selected after addressing such items as:

- Depth of waterbody
- Depth of sample
- Stratification
- Seasonal variations
- Analytical parameters of interest.

The following general procedure should be used to obtain representative surface water and leachate seep samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to sampling: date and time, sample method, and sample depth.

- Collect the sample from the surface water, within several tenths of a foot of the streambed, by immersing a new, dedicated 1-L glass or volatile organic analyte sample container into the waterbody. If a stream is being sampled, collect the sample upstream of the sampler with the opening of the sampling device oriented upstream but avoiding floating debris.

- Directly fill the appropriate sample containers from the 1-L or volatile organic analyte sampling device.

- Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- Water sample containers are generally filled directly from the source or sampler without special considerations. The exception is the collection of aqueous VOC samples requiring pH adjustment. VOC samples will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation methods are discussed in Section 3.6.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- Measure water quality indicator parameters, if possible, by direct immersion of instrument probes into the waterbody immediately following sample collection. If direct measurement is not possible, measure these parameters from water remaining in the sampling device or another sample bottle. Record this information in the field logbook and sample data record.

- Complete remaining portions of the Field Record of Surface Water and Sediment Sampling form (Figure SOP048-2) after each station is sampled, including: time of sample collection, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

3.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

  — Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the 1-L container, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

3.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the surface water and leachate seep samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.
- Equipment (Rinsate) Blank—Required at a frequency of once per day per media sampled.
- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

3.7 DECONTAMINATION

Field monitoring equipment will be decontaminated prior to use and following sampling of each station by the procedure listed below. Laboratory pre-cleaned, dedicated 1-L glass sample collection containers are used once and discarded and, therefore, do not undergo any decontamination. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

4. REFERENCES

U.S. Environmental Protection Agency. 1996. Groundwater Issue-Low Flow Sampling (Minimal Drawdown) Groundwater Sampling Procedures. April.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:		Project Number:	
Well ID:		Well Lock Status:	
Well Condition:		Weather:	

Gauge Date:		Gauge Time:	
Sounding Method:		Measurement Ref:	
Stick Up/Down (ft):		Well Diameter (in.):	

Purge Date:		Purge Time:	
Purge Method:		Field Personnel:	
Ambient Air VOCs (ppm):		Well Mouth VOCs (ppm):	

WELL VOLUME

A. Well Depth (ft):		D. Well Volume/ft (L):	
B. Depth to Water (ft):		E. Well Volume (L) (C*D):	
C. Liquid Depth (ft) (A-B)		F. Three Well Volumes (L) (E*3):	

G. Measurable LNAPL? Yes _____ /ft No _____

Parameter	Beginning	1	2	3	4	5
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Total Quantity of Water Removed (L):			
Samplers:		Sampling Time (Start/End):	
Sampling Date:		Decontamination Fluids Used:	
Sample Type:		Sample Preservatives:	
Sample Bottle IDs:			
Sample Parameters:			

Figure SOP048-1.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:	Project Number:	Date:
Well ID:	Field Personnel:	

Parameter	6	7	8	9	10	11
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Parameter	12	13	14	15	16	17
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Comments and Observations:

Figure SOP048-1.

FIELD RECORD OF SURFACE WATER AND SEDIMENT SAMPLING

Site Name:			Project Number:
Sample Location ID:			Date:
Time:	Start:	End:	Sample Team Members:

SURFACE WATER INFORMATION

Type of Surface Water:
( ) Stream ( ) River
( ) Pond/Lake ( ) Seep

Water Depth and Sample Location _____ (ft)

Depth of Sample from Top of Water _____ (ft)

Equipment Used for Collection:
( ) None, Grab into Bottle
( ) Bomb Sampler
( ) Pump _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Water Quality Parameters
( ) Temperature ____
( ) Conductivity ___ µmhs/cm
( ) pH ____ units
( ) Dissolved oxygen ____ mg/L
( ) Turbidity ____ NTU
( ) Eh ____ mv

Velocity Measurements Obtained? ( ) No ( ) Yes, See Flow Measurement Data Record

Field QC Data: ( ) Duplicate ID _____ Used:
( ) MS/MSD

Field Duplicate Collected
( ) Yes
( ) No

Sample Location Sketch: Method
( ) Winkler
( ) Probe

SEDIMENT INFORMATION

Type of Sample Collected:
( ) Discrete
( ) Composite

Sediment Type:
( ) Clay
( ) Sand
( ) Organic
( ) Gravel

Equipment Used for Collection:
( ) Gravity Corer
( ) Stainless Steel Split Spoon
( ) Dredge
( ) Hand Spoon/Trowel
( ) Aluminum Pans
( ) Stainless Steel Bucket
( ) _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Sample Observations:
( ) Odor _____
( ) Color

Field QC Data: ( ) Field Duplicate Collected ( ) MS/MSD
Duplicate ID _____

SAMPLES COLLECTED

Check if Required at this Location	Matrix: Surface Water	Matrix: Sediment	Check if Preserved with Acid/Base	Volume Required	Check if Sample Collected	Sample Bottle IDs			

NOTES/SKETCH

Figure SOP048-2.

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COVER PAGE

Standard Operating Procedure:
Sampling Ground Water With a HydraSleeve (patents #6,481,300; 6,837,120)

Introduction

The HydraSleeve is classified as a no-purge (passive) grab sampling device, meaning that it is used to collect ground-water samples directly from the screened interval of a well without having to purge the well prior to sample collection. When it is used as described in this Standard Operating Procedure (SOP), the HydraSleeve causes no drawdown in the well (until the sample is withdrawn from the water column) and only minimal disturbance of the water column, because it has a very thin cross section and it displaces very little water (<100 ml) during deployment in the well. The HydraSleeve collects a sample from within the screen only, and it excludes water from any other part of the water column in the well through the use of a self-sealing check valve at the top of the sampler. It is a single-use (disposable) sampler that is not intended for reuse, so there are no decontamination requirements for the sampler itself.

The use of no-purge sampling as a means of collecting representative ground-water samples depends on the natural movement of ground water (under ambient hydraulic head) from the formation adjacent to the well screen through the screen. Robin and Gillham (1987) demonstrated the existence of a dynamic equilibrium between the water in a formation and the water in a well screen installed in that formation, which results in formation-quality water being available in the well screen for sampling at all times. No-purge sampling devices like the HydraSleeve collect this formation-quality water as the sample, under undisturbed (non-pumping) natural flow conditions. Samples collected in this manner generally provide more conservative (i.e., higher concentration) values than samples collected using well-volume purging (which are generally considered to be more representative of actual ground-water chemistry), and values equivalent to samples collected using low-flow purging and sampling (Parsons, 2005).

Applications of the HydraSleeve

The HydraSleeve can be used to collect representative samples of ground water for all analytes (volatile organic compounds [VOCs], semi-volatile organic compounds [SVOCs], common metals, trace metals, major cations and anions, dissolved gases, total dissolved solids, radionuclides, pesticides, PCBs, explosive compounds, and all other analytical parameters). Designs are available to collect samples from wells from 1" inside diameter and larger. The HydraSleeve can collect samples from wells of any yield, but it is especially well-suited to collecting samples from low-yield wells, where other sampling methods can't be used reliably because their use results in dewatering of the well screen and alteration of sample chemistry (McAlary and Barker, 1987).

The HydraSleeve can collect samples from wells of any depth, and it can be used for single-event sampling or long-term ground-water monitoring programs. Because of its thin cross section and flexible construction, it can be used in narrow, constricted or damaged wells where rigid sampling devices may not fit. Using multiple HydraSleeves deployed in series along a

single suspension line or tether, it is also possible to conduct in-well vertical profiling in wells in which contaminant concentrations are thought to be stratified.

HydraSleeves should not be used to collect ground-water samples from wells in which separate (non-aqueous) phase hydrocarbons (i.e., gasoline, diesel fuel or jet fuel) are present because of the possibility of incorporating some of the separate-phase hydrocarbon into the sample.

Description of the HydraSleeve

The HydraSleeve (Figure 1) consists of a the following basic components:

- A suspension line or tether (A.), attached to the spring clip or directly to the top of the sleeve to deploy the device into and recover the device from the well. Tethers with depth indicators marked in 1-foot intervals are available from the manufacturer.
- A long, flexible, 4-mil thick lay-flat polyethylene sample sleeve (C.) sealed at the bottom (this is the sample chamber), which comes in different sizes, as discussed below with a self-sealing reed-type flexible polyethylene check valve built into the top of the sleeve (B.) to prevent water from entering or exiting the sampler except during sample acquisition.
- A reusable stainless-steel weight with clip (D.), which is attached to the bottom of the sleeve to carry it down the well to its intended depth in the water column. Bottom weights available from the manufacturer are 0.75" OD and are available in three sizes: 5 oz. (2.5" long); 8 oz. (4" long); and 16 oz. (8" long). In lieu of a bottom weight, an optional top weight may be attached to the top of the HydraSleeve to carry it to depth and to compress it at the bottom of the well (not shown in Figure 1);
- A discharge tube that is used to puncture the HydraSleeve after it is recovered from the well so the sample can be decanted into sample bottles (not shown).
- Just above the self-sealing check valve at the top of the sleeve are two holes which provide attachment points for the spring clip and/or suspension line or tether. At the bottom of the sample sleeve are two holes which provide attachment points for the weight clip and weight.

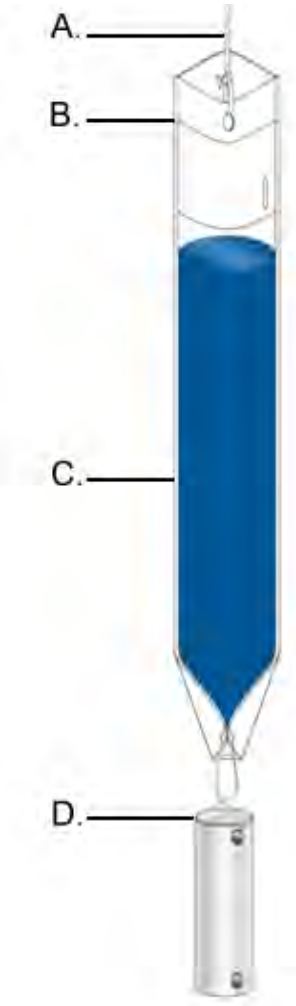

Figure 1. HydraSleeve components.

Note: The sample sleeve and the discharge tube are designed for one-time use and are disposable. The spring clip, weight and weight clip may be reused after thorough cleaning. Suspension cord is generally disposed after one use although, if it is dedicated to the well, it may be reused at the discretion of the sampling personnel.

Selecting the HydraSleeve Size to Meet Site-Specific Sampling Objectives

It is important to understand that each HydraSleeve is able to collect a finite volume of sample because, after the HydraSleeve is deployed, you only get one chance to collect an undisturbed sample. Thus, the volume of sample required to meet your site-specific sampling and analytical requirements will dictate the size of HydraSleeve you need to meet these requirements.

The volume of sample collected by the HydraSleeve varies with the diameter and length of the HydraSleeve. Dimensions and volumes of available HydraSleeve models are detailed in Table 1.

Table 1. Dimensions and volumes of HydraSleeve models.

Diameter	Volume	Length	Lay-Flat Width	Filled
2-Inch HydraSleeves				
Standard 625-ml HydraSleeve	625 ml	30”	2.5”	1.4”
Standard 1-Liter HydraSleeve	1 Liter	38”	3”	1.9”
1-Liter HydraSleeve SS	1 Liter	36”	3”	1.9”
2-Liter HydraSleeve SS	2 Liters	60”	3”	1.9”
4-Inch HydraSleeves				
Standard 1.6-Liter HydraSleeve	1.6 Liters	30”	3.8”	2.3”
Custom 2-Liter HydraSleeve	2 Liters	36”	4”	2.7”

HydraSleeves can be custom-fabricated by the manufacturer in varying diameters and lengths to meet specific volume requirements. HydraSleeves can also be deployed in series (i.e., multiple HydraSleeves attached to one tether) to collect additional sample to meet specific volume requirements, as described below.

If you have questions regarding the availability of sufficient volume of sample to satisfy laboratory requirements for analysis, it is recommended that you contact the laboratory to discuss the minimum volumes needed for each suite of analytes. Laboratories often require only 10% to 25% of the volume they specify to complete analysis for specific suites of analytes, so they can often work with much smaller sample volumes that can easily be supplied by a HydraSleeve.

HydraSleeve Deployment

Information Required Before Deploying a HydraSleeve

Before installing a HydraSleeve in any well, you will need to know the following:

- The inside diameter of the well;
- The total depth of the well;
- The position of the well screen in the well;
- The length of the well screen; and
- The water level in the well.

The inside diameter of the well is used to determine the appropriate HydraSleeve diameter for use in the well. The other information is used to determine the proper placement of the HydraSleeve in the well to collect a representative sample from the screen (see HydraSleeve Placement, below), and to determine the appropriate length of tether to attach to the HydraSleeve to deploy it at the appropriate position in the well.

Most of this information (with the exception of the water level) should be available from the well log; if not, it will have to be collected by some other means. The inside diameter of the well can be measured at the top of the well casing, and the total depth of the well can be measured by sounding the bottom of the well with a weighted tape. The position and length of the well screen may have to be determined using a down-hole camera if a well log is not available. The water level in the well can be measured using any commonly available water-level gauge.

HydraSleeve Placement

The HydraSleeve is designed to collect a sample directly from the well screen, and it fills by pulling it up through the screen a distance equivalent to 1 to 1.5 times its length. This upward motion causes the top check valve to open, which allows the device to fill. To optimize sample recovery, it is recommended that the HydraSleeve be placed in the well so that the bottom weight rests on the bottom of the well and the top of the HydraSleeve is as close to the bottom of the well screen as possible. This should allow the sampler to fill before the top of the device reaches the top of the screen as it is pulled up through the water column, and ensure that only water from the screen is collected as the sample. In short-screen wells, or wells with a short water column, it may be necessary to use a top-weight on the HydraSleeve to compress it in the bottom of the well so that, when it is recovered, it has room to fill before it reaches the top of the screen.

Example

2" ID PVC well, 50' total depth, 10' screen at the bottom of the well, with water level above the screen (the entire screen contains water).

Correct Placement (figure 2): Using a standard HydraSleeve for a 2" well (2.6" flat width/1.5" filled OD x 30" long, 650 ml volume), deploy the sampler so the weight (an 8 oz., 4"-long weight with a 2"-long clip) rests at the bottom of the well. The top of the sleeve is thus set at about 36" above the bottom of the well. When the sampler is recovered, it will be pulled upward approximately 30" to 45" before it is filled; therefore, it is full (and the top check valve closes) at approximately 66" (5 ½ feet) to 81" (6 ¾ feet) above the bottom of the well, which is well before the sampler reaches the top of the screen. In this example, only water from the screen is collected as a sample.

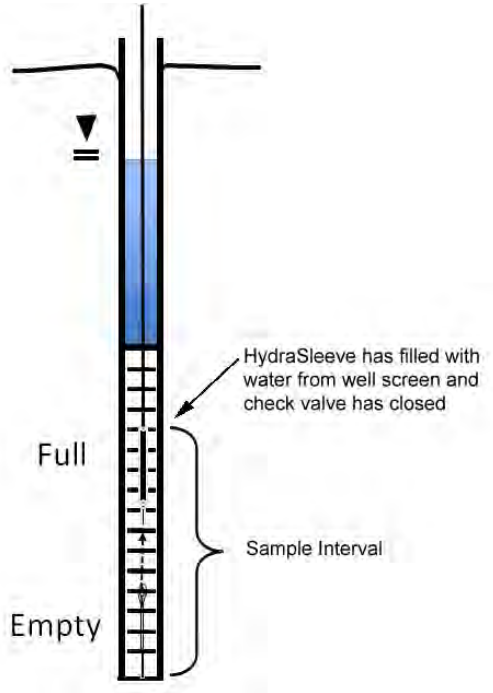

Figure 2. Correct placement of HydraSleeve.

Incorrect Placement (figure 3): If the well screen in this example was only 5' long, and the HydraSleeve was placed as above, it would not fill before the top of the device reached the top of the well screen, so the sample would include water from above the screen, which may not have the same chemistry.

The solution? Deploy the HydraSleeve with a top weight, so that it is collapsed to within 6" to 9" of the bottom of the well. When the HydraSleeve is recovered, it will fill within 39" (3 ¼ feet) to 54" (4 ½ feet) above the bottom of the well, or just before the sampler reaches the top of the screen, so it collects only water from the screen as the sample.

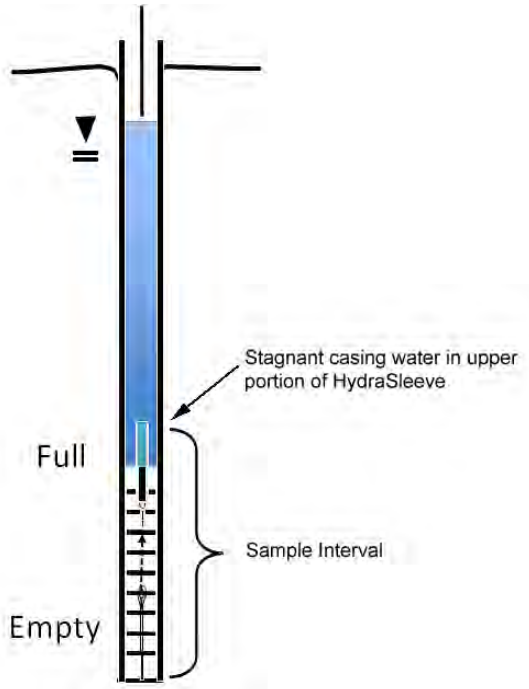

Figure 3. Incorrect placement of HydraSleeve.

Procedures for Sampling with the HydraSleeve

Collecting a ground-water sample with a HydraSleeve is a simple one-person operation.

Note: Before deploying the HydraSleeve in the well, collect the depth-to-water measurement that you will use to determine the preferred position of the HydraSleeve in the well. This measurement may also be used with measurements from other wells to create a ground-water contour map. If necessary, also measure the depth to the bottom of the well to verify actual well depth to confirm your decision on placement of the HydraSleeve in the water column.

Measure the correct amount of tether needed to suspend the HydraSleeve in the well so that the weight will rest on the bottom of the well (or at your preferred position in the well). Make sure to account for the need to leave a few feet of tether at the top of the well to allow recovery of the sleeve

Note: Always wear sterile gloves when handling and discharging the HydraSleeve.

I. Assembling the HydraSleeve

1. Remove the HydraSleeve from its packaging, unfold it, and hold it by its top.

2. Crimp the top of the HydraSleeve by folding the hard polyethylene reinforcing strips at the holes.

3. Attach the spring clip to the holes to ensure that the top will remain open until the sampler is retrieved.

4. Attach the tether to the spring clip by tying a knot in the tether.

Note: Alternatively, attach the tether to one (NOT both) of the holes at the top of the Hydrasleeve by tying a knot in the tether.

5. Fold the flaps with the two holes at the bottom of the HydraSleeve together and slide the weight clip through the holes.

6. Attach a weight to the bottom of the weight clip to ensure that the HydraSleeve will descend to the bottom of the well.

II. Deploying the HydraSleeve

1. Using the tether, carefully lower the HydraSleeve to the bottom of the well, or to your preferred depth in the water column

   During installation, hydrostatic pressure in the water column will keep the self-sealing check valve at the top of the HydraSleeve closed, and ensure that it retains its flat, empty profile for an indefinite period prior to recovery.

> **Note:** Make sure that it is not pulled upward at any time during its descent. If the HydraSleeve is pulled upward at a rate greater than 0.5'/second at any time prior to recovery, the top check valve will open and water will enter the HydraSleeve prematurely.

2. Secure the tether at the top of the well by placing the well cap on the top of the well casing and over the tether as the tether is held taut.

> **Note:** Alternatively, you can ty the tether to a hook on the bottom of the well cap (you will need to leave a few inches of slack in the line to avoid pulling the sampler up as the cap is removed at the next sampling event).

III. Equilibrating the Well

The equilibration time is the time it takes for conditions in the water column (primarily flow dynamics and contaminant distribution) to restabilize after vertical mixing occurs (caused by installation of a sampling device in the well).

- Situation: The HydraSleeve is deployed for the first time or for only one time in a well

  The HydraSleeve is very thin in cross section and displaces very little water (<100 ml) during deployment so, unlike most other sampling devices, it does not disturb the water column to the point at which long equilibration times are necessary to ensure recovery of a representative sample.

  In most cases, the HydraSleeve can be recovered immediately (with no equilibration time) or within a few hours. In regulatory jurisdictions that impose specific requirements for equilibration times prior to recovery of no-purge sampling devices, these requirements should be followed.

- Situation: The HydraSleeve is being deployed for recovery during a future sampling event

  In periodic (i.e., quarterly or semi-annual) sampling programs, the sampler for the current sampling event can be recovered and a new sampler (for the next sampling event)

deployed immediately thereafter, so the new sampler remains in the well until the next sampling event.

Thus, a long equilibration time is ensured and, at the next sampling event, the sampler can be recovered immediately. This means that separate mobilizations, to deploy and then to recover the sampler, are not required. HydraSleeves can be left in a well for an indefinite period of time without concern.

IV. HydraSleeve Recovery and Sample Collection

1. Hold on to the tether while removing the well cap.

2. Secure the tether at the top of the well while maintaining tension on the tether (but without pulling the tether upwards)

3. Measure the water level in the well.

4. In one smooth motion, pull the tether up between 30" to 45" (36" to 54" for the longer HydraSleeve) at a rate of about 1' per second (or faster).

   The motion will open the top check valve and allow the HydraSleeve to fill (it should fill in about 1 to 1.5 times the length of the HydraSleeve). This is analogous to coring the water column in the well from the bottom up.

   When the HydraSleeve is full, the top check valve will close. You should begin to feel the weight of the HydraSleeve on the tether and it will begin to displace water. The closed check valve prevents loss of sample and entry of water from zones above the well screen as the HydraSleeve is recovered.

5. Continue pulling the tether upward until the HydraSleeve is at the top of the well.

6. Decant and discard the small volume of water trapped in the Hydrasleeve above the check valve by turning the sleeve over.

V. Sample Collection

Note: Sample collection should be done immediately after the HydraSleeve has been brought to the surface to preserve sample integrity.

1. Remove the discharge tube from its sleeve.

2. Hold the HydraSleeve at the check valve.

3. Puncture the HydraSleeve just below the check valve with the pointed end of the discharge tube

4. Discharge water from the HydraSleeve into your sample containers.

Control the discharge from the HydraSleeve by either raising the bottom of the sleeve, by squeezing it like a tube of toothpaste, or both.

5. Continue filling sample containers until all are full.

Measurement of Field Indicator Parameters

Field indicator parameter measurement is generally done during well purging and sampling to confirm when parameters are stable and sampling can begin. Because no-purge sampling does not require purging, field indicator parameter measurement is not necessary for the purpose of confirming when purging is complete.

If field indicator parameter measurement is required to meet a specific non-purging regulatory requirement, it can be done by taking measurements from water within a HydraSleeve that is not used for collecting a sample to submit for laboratory analysis (i.e., a second HydraSleeve installed in conjunction with the primary sample collection HydraSleeve [see Multiple Sampler Deployment below]).

Alternate Deployment Strategies

Deployment in Wells with Limited Water Columns

For wells in which only a limited water column exists to be sampled, the HydraSleeve can be deployed with an optional top weight instead of a bottom weight, which collapses the HydraSleeve to a very short (approximately 6” to 9”) length, and allows the HydraSleeve to fill in a water column only 36” to 45” in height.

Multiple Sampler Deployment

Multiple sampler deployment in a single well screen can accomplish two purposes:

- It can collect additional sample volume to satisfy site or laboratory-specific sample volume requirements.
- It can accommodate the need for collecting field indicator parameter measurements.
- It can be used to collect samples from multiple intervals in the screen to allow identification of possible contaminant stratification.

It is possible to use up to 3 standard 30" HydraSleeves deployed in series along a single tether to collect samples from a 10' long well screen without collecting water from the interval above the screen.

The samplers must be attached to the tether with the first (attached to the tether as described above, and with the weight at the bottom) at the bottom of the screen, the second (attached to the tether using a cable tie or stainless-steel clip available from the manufacturer) immediately above the first, and the third (attached the same as the second) immediately above the second.

Alternately, the first sampler can be attached to the tether as described above, a second attached to the bottom of the first using a short length of tether (in place of the weight), and the third attached to the bottom of the second in the same manner, with the weight attached to the bottom of the third sampler.

In either case, when attaching multiple HydraSleeves in series, more weight may be required to hold the samplers in place in the well than would be required with a single sampler. Recovery of multiple samplers and collection of samples is done in the same manner as for single sampler deployments.

Post-Sampling Activities

The recovered HydraSleeve and the sample discharge tubing should be disposed as per the solid waste management plan for the site. To prepare for the next sampling event, a new HydraSleeve can be deployed in the well (as described previously) and left in the well until the next sampling event, at which time it can be recovered.

The weight and weight clip can be reused on this sampler after they have been thoroughly cleaned as per the site equipment decontamination plan. The tether may be reused or discarded at the discretion of sampling personnel.

References

McAlary, T. A. and J. F. Barker, 1987, Volatilization Losses of Organics During Ground-Water Sampling From Low-Permeability Materials, Ground-Water Monitoring Review, Vol. 7, No. 4, pp. 63-68

Parsons, 2005, Results Report for the Demonstration of No-Purge Ground-Water Sampling Devices at Former McClellan Air Force Base, California; Contract F44650-99-D-0005, Delivery Order DKO1, U.S. Army Corps of Engineers (Omaha District), U.S. Air Force Center for Environmental Excellence, and U.S. Air Force Real Property Agency

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VAPOR INTRUSION SAMPLING PROCEDURES

1. INTRODUCTION

An inspection of general site conditions will be performed at each property location as part of the air sampling. The inspection will include the following activities:

- Completion of an Indoor Air Quality Questionnaire and Building Inventory (attached).
- Documentation of outdoor weather conditions and indoor temperature.
- Selection of air sampling locations.

1.1 FIELD ACTIVITIES

The semi-permanent, sub-slab soil gas monitoring points will be installed prior to the collection of indoor and outdoor air samples so that sampling activities can occur within the same timeframe as close to simultaneously as possible. Depending on the type of media used to seal the monitoring point, each semi-permanent sub-slab soil gas monitoring point will be allowed to set up for at least 12 hours prior to purging and leak check testing. Once the purging and leak check tests have been performed, indoor air and outdoor ambient air samples will be set up for sample collection. The indoor and outdoor air samples will be collected concurrently over the same time period as the sub-slab samples. Subsequent rounds of sampling will follow the same protocol as implemented during the first round, so that analytical data results from both sampling events will be comparable.

During field activities, field data sheets will be used to record details of the sub-slab soil gas, indoor air, and outdoor ambient air sampling activities, which will include the following information, at a minimum and as applicable:

- Source area identification and building identification
- Sample identification
- Sampling location
- Sub-slab monitoring point installation date and time
- Summa canister and associated flow-control device serial number identification
- Flow-control device sample flow rate (set by laboratory)
- Initial Summa canister pressure using hand-held vacuum gauge
- Summa canister sample duration
- Helium concentration in shroud during leak check testing
- Photoionization detector (PID) reading from sub-slab monitoring point purge
- Significant comments and notes during installation or sample collection activities.

At each location, air samples will be collected for laboratory analysis utilizing the appropriate U.S. Environmental Protection Agency method. Air samples will be collected from two locations per structure, including the first floor and the sub-slab environment. If a basement is present, air samples will be collected from three locations per structure, including the first floor, basement, and sub-slab environment. An active approach, utilizing

laboratory batch-certified Summa canisters regulated for 24-hour sample collection, will be used based on building use and/or project requirements to evaluate the indoor air and sub-slab soil vapor conditions.

1.2 SUMMA CANISTERS

Six-liter (L) Summa canisters will be used for the collection of air samples. The rationale for this recommendation includes the following:

The analysis of VOCs by TO-15, TO-15 selective ion monitoring (SIM), and/or fixed gases by ASTM D-1946 using a smaller volume (1-L) Summa canister requires that the entire sample volume be used for analysis. Therefore, additional sample volume is not available for confirmation analysis should additional quantification be required.

The dilution factor is lower for 6-L Summa canisters because they are pressurized to 5 pounds per square inch (psi) to retrieve the analytical aliquot, resulting in an expected dilution factor of 1.61 (compared to an expected dilution factor of 2.42 for 1-L Summa canisters, which are pressurized to 15 psi).

The Summa canisters will be paired with flow regulators that will be used at only one location, and then set aside for return to the laboratory for decontamination. The flow regulator, which features a particulate filter and vacuum gauge, will be set to a specified flow rate by the analytical laboratory prior to shipping. The Summa canisters used during this project will be individually certified by the analytical laboratory, and set at a vacuum pressure of approximately negative 30 inches of mercury (in. Hg). Immediately prior to sample collection, the vacuum for each Summa canister will be verified with a handheld pressure gauge to assess for tightness during transit from the laboratory to the field. Before removing the compression plug on the canister during the pressure check, confirm the canister valve is closed and did not come open during shipping. Pressure readings will be recorded on the field data forms and tags. Any Summa canister with a pressure reading less than -25 in. Hg will not be used.

2. SUB-SLAB SAMPLING PROCEDURES

2.1 SUB-SLAB MONITOR POINT INSTALLATION

The procedures for the selection and installation of sub-slab soil gas sampling points within buildings are based on applicable guidance. The procedures are intended to provide the specific process used to select and install sample sub-slab soil gas monitoring probes. However, the procedures are also intended to be flexible enough to implement within a variety of buildings and using different materials, based on local availability. The procedures presented below will be followed for the selection and installation of each sub-slab soil gas monitoring point within the buildings to be sampled during the field investigation:

1. Features that tend to be preferential migration pathways for vapors (utility banks, piping corridors, etc.) will be identified and samples will be taken in the vicinity of, but no

closer than 6 feet (ft) from, these features, if present. A visual assessment of the condition of the floor will also be completed. If a utility clearance determines that the sub-slab soil gas monitoring points cannot occur at the proposed location, then location will be adjusted to the closest practicable point. Sub-slab soil gas sampling locations will be selected to be out of the line of traffic and will be placed a minimum of 6 ft from an exterior wall and/or major cracks in the concrete slab.

2. Once the sampling points are located, semi-permanent sub-slab soil gas monitoring point will be installed to allow for multiple sampling events from identical sub-slab locations. The semi-permanent sub-slab soil gas monitoring point will be installed by drilling a 1-in.-diameter hole into the concrete slab to an approximate depth of 1½ to 2 in. using a hand-held electric hammer drill or equivalent to create an annular space for the semi-permanent point. A ¼-in.-diameter drill bit will then be used in the center of the 1-in.-diameter opening to create a borehole through the remainder of the concrete slab to approximately 2 in. into the underlying material.

3. After drilling through the slab, record the approximate thickness of the slab and clean any dust from the slab and wipe with a dampened towel. A shop vacuum can be used to clear the 1-in.-diameter borehole of loose material before drilling all the way through the slab. Do not vacuum the open hole, because vapors could be transmitted from under the slab into the indoor air that would bias the indoor air sample.
4. Upon completion of the borehole, an appropriate length of ¼-in.-outside diameter (OD) stainless-steel tubing will be cut to extend from ½ inch above the bottom of the 1-in.-diameter annular space borehole to a depth approximately 1 in. below the bottom of the slab. The stainless-steel tubing will then be attached to a ¼-in. stainless-steel union and tightened to secure the tubing in place. The assembly will then be placed into the borehole and checked for tightness within the borehole. The sub-slab soil gas monitoring point will also be equipped with a ¼-in. stainless-steel threaded plug so that it may be sealed from ambient air intrusion and/or sub-slab soil gas leakage to indoor air. The semi-permanent point should be recessed slightly under the surface of the concreate to prevent a tripping hazard.

5. The bottom of the stainless-steel tube will be set at approximately 1 to 2 in. below the concrete slab surface; therefore, the intake interval will be located within the near sub-slab environment, targeting the likeliest source of near-slab soil gas. Caution should be taken not to plug the end of the tube with the underlying material during installation.

6. A thin layer (approximately ¼ in. thick) of melted beeswax will be placed at the base of the 1-in.-diameter annular space borehole to seal the tubing and any potential void spaces between the stainless-steel tube and the ¼-in.-diameter borehole. The beeswax will be allowed to air cool and harden in place.

7. Following beeswax placement and cooling, a ½ to ¾-in.-thick (up to the lowest threads on the union) layer of quick-set expansion-type concrete will be placed into the 1-in.-diameter annular space borehole to cover the beeswax and further seal the sub-slab soil gas monitoring point preventing air communication between the sub- slab environment and the indoor air space.

8. After the sub-slab soil gas monitoring point is installed, the stainless-steel plug will be hand-tightened until snug, being careful not to over-tighten to avoid breaking the concrete seal.

9. The sub-slab soil gas monitoring points will be allowed to set up prior to conducting leak-check testing, purging, and sampling activities.

2.2 SUB-SLAB MONITOR POINT LEAK TEST PROCEDURE

Ambient air intrusion into gas/air samples may result in a dilution of the gas/air sample, and may produce results that underestimate actual site concentrations; or alternatively, may contaminate the sample with aboveground indoor air contaminants. Leak tests will be conducted at each sub- slab soil gas monitoring point. The leak tests to be employed during the field activities include a shut-in leak test and a tracer leak test. These leak tests will be used to assess whether a good seal was established in the sampling train, ground surface, and the probe interface. A leak can be considered present when the tracer compound is present in the test sample at more than 10 percent of the source concentration.

The tracer test is also designed to check for leaks in aboveground fittings, as well as the sub-slab soil gas monitoring point surface seal interface. The monitoring point seal integrity will be confirmed in real time by analyzing soil gas purge samples for the selected tracer compound. Helium will be used as the tracer compound during field activities; however, other tracer compounds, such as pentane, isopropanol, isobutene, propane, or butane, may be acceptable for use if the selected tracer compound is not a chemical of potential concern (COPC). Additional detail on leak test implementation is provided below. Additional or alternate leak detection methods may be acceptable if fully documented during field implementation.

2.2.1 Sub-Slab Sample Train Shut-In Test

Before connecting the flow regulator, check the Summa canister initial pressure and verify that the canister valve opens and closes correctly during the initial pressure check. A ¼-in. stainless- steel sample train will be connected to the flow regulator using compression fittings. The stainless-steel sample train will consist of a stainless-steel union tee with an attached stainless- steel ball valve located near the connection to the flow regulator for purging of the sample train. At the opposite end of the stainless-steel sample train, attach ¼-in. Teflon or Teflon-lined tubing (less than 3 ft) from the end of stainless-steel sample train to the sampling port for flexibility.

After the Teflon tubing is attached to the sample train, a shut-in test can be performed to check the connections at the flow regulator and along the sample train. With the purge valve closed, attach a hand vacuum pump to the end of the Teflon tube and apply a vacuum between -10 to -20 in. Hg on the sample train. If the vacuum does not drop over a 1-minute time interval, then the sample train is considered to be leak-free. If the vacuum does start dropping, then one of the connections is compromised and may need to be tightened and rechecked. If the sample connections cannot be determined to be leak-free, the use of another canister and flow regulator or sample train should be considered. Leaks are typically common on connections between flow regulators and canisters because of continuous reuse and possible over-tightening can

damage connections.

After the shut-in test is complete, remove the hand vacuum pump and the Teflon tubing inserted through the ¼-inch opening at the top of the leak test shroud; then connect to the semi-permanent monitoring point installed in the slab. The shroud is then slid down the tubing to create an air chamber above the monitoring point.

2.2.2 Sub-Slab Sample Train Purging

Prior to collection of sub-slab soil gas samples, the sub-slab soil gas monitoring points will be purged of vapor using the calibrated low-flow purge pump to remove approximately three pore volumes from the sampling zone. The pore volume for the sub-slab soil gas monitoring points will be calculated based on known diameter and length, with an assumed 25-percent porosity, plus the internal volume of the tubing. The purge volume is typically 1 L, or about one Tedlar bag. The purpose of the purge is to ensure stagnant or ambient air is removed from the sampling system prior to sample collection. The purge will be completed at the same time as the leak test is performed. Purge volumes will be kept to a minimum to decrease the chance of leakage, reduce additional partitioning of potential contaminants into the vapor phase, and unnecessary movement of the soil gas to the sampling probe.

To complete the purge, the purge pump will be connected to the stainless-steel sample train near the canister at the ball valve using a short length of Teflon tubing with flexible Tygon tubing. The internal length of the Tygon tubing will be minimized whenever possible by fully inserting both ends of the tubing being connected. The outlet of the purge pump (Gil Air5 or similar) will be connected to a Tedlar bag using Teflon tubing connected with short lengths of flexible tubing. When purging is complete, the valve should be closed before turning the pump off to insure indoor air is not allowed into the sample train.

2.2.3 Sub-Slab Sample Port Tracer Gas Test

The final portion of the test is to validate the connection of the sample train to the sub-slab monitoring point and the seal of the monitoring point at the concrete slab. During the purge, medical-grade helium tracer gas will be applied directly to a shroud or "bucket" covering the sub-slab soil gas monitoring point by directing a tube from a helium tank source into the shroud. The interior space of the shroud will be monitored for helium concentration using a Radiodetection Helium/Hydrogen Multi-Gas Detector, Model MGD-2002. Once the interior of the shroud reaches approximately 50-percent helium, the purge pump will be activated and allowed to purge approximately 1 L of volume into the Tedlar bag from the sub-slab soil gas monitoring point. Slight pressure should be placed on the top of the shroud to create a seal at the shroud and concrete interface until the purge is complete. At this time, the helium detector will be removed from the shroud and the shroud plugged to retain the helium concentration. The helium detector will be allowed to equilibrate with atmosphere and later be connected to the Tedlar bag to assess helium concentration. The monitoring point will be considered sealed from atmospheric air intrusion if the helium meter does not detect 5 percent helium in the Tedlar bag. Additionally, the remaining volume of the Tedlar bag can be used to screen sub-slab soil gas concentrations of volatile organic compounds (VOCs) using a ppbRae or equivalent

PID. All leak test and purge information should be recorded on the field sample form.

If helium is detected in the Tedlar bag above 5 percent, the integrity of the monitoring point will be assessed and repaired, if possible. Modeling clay may be utilized to seal potential cracks or penetrations in the monitoring point vault. Following confirmation that the monitoring point vault has been sealed from atmospheric air intrusion, the purge process should begin again. If the monitoring point vault is unable to be sealed, it should be abandoned and the location restored to pre-sample conditions. A replacement monitoring point should be installed at least 5 ft away from the initial location.

All sub-slab locations should be leak tested before activating the Summa canisters at other locations in the building. This ensures that if problems are encountered during leak testing, the sampling port can be reinstalled. Sampling should be postponed an additional day to allow for the newly-installed port to set up and ensure that vapor intrusion sample collection occurs within the same timeframe for that building.

2.3 SUB-SLAB SAMPLING PROCEDURE

After the leak test has been performed on the sample train and sampling ports for the building, the purge valve on the sample train should be checked to make sure it is in the closed position. As the Summa canister is turned on for sample collection, the regulator pressure should be checked against the initial pressure recorded on the canister sample tag. If there is a major difference in pressure, lightly tap on the gauge to ensure it is not sticking. The pressure, date, start time, and serial number of the canister and associated regulator should be recorded on the field sample form or in a field logbook along with the sample identification and any other important information. Typically an indoor air sample is co-located with the sub-slab location and would be initiated concurrently.

The canister setup and the surrounding area should be photographed. The next sub-slab location should be started within a few minutes following the first location to allow end times to stagger for shutdown of canisters. The Summa canister pressure should be checked periodically and gauges should tapped upon reading to determine if pressure gauge is reading correctly. The Summa canister should be closed at the flow regulator designated 24-hour sample time if the appropriate sample volume is collected. Sample volume depends on the amount the laboratory needs to run a sample and may be differ between laboratories. In most cases, if the initial canister pressure is at -30 in. Hg, then a sufficient sample volume would be achieved when the pressure was at -10 in. Hg.

The Summa canister should be closed if internal pressure reaches -5 in. Hg before the designated sample time. This will ensure that the Summa canister is shipped back to the lab under a low negative pressure for sample quality control. The Summa canister stop time will be used as the sample time on the chain-of-custody form. The Summa canister and regulator should be disassembled and packaged together during shipping. All information on the tag and field sampling form should be filled out along with the chain-of-custody form.

A duplicate sub-slab sample can be collected using a duplicate sampling tee. Two Summa canisters with flow regulators can be connected together using a sampling tee with equal lengths that come together to connect into the sampling train. Both canisters are usually started and stopped at the same time even when they differ in ending pressure.

3. INDOOR AMBIENT AIR SAMPLING PROCEDURES

The following procedures for the collection of indoor air samples are based on applicable guidance and practical experience during the implementation of field activities on other projects. The procedures are intended to provide a specific process to follow for the collection of indoor air and outdoor ambient air samples. However, the procedures are also intended to be flexible enough to implement based on conditions encountered in the field.

Indoor air and outdoor ambient air samples will be collected at each building retained for additional evaluation. Potential indoor air sources of COPCs remaining in buildings, including the location within the building(s) and type of potential impact, will be documented. Portable vapor monitoring equipment may be useful for identifying unknown sources within buildings.

The operation of heating, ventilation, and air conditioning (HVAC) systems during sample collection will be noted on the Building Inventory and Indoor Air Sampling Questionnaire. When sampling activities are conducted, the building HVAC system should be operating in a manner consistent with normal operating conditions during building occupation.

To minimize the potential for interferences or dilution of indoor air samples, occupants will be asked to make a reasonable effort to avoid the following for a minimum of 24 hours prior to sampling and during the sample collection process:

- Opening windows or vents.
- Operating ventilation fans (unless special arrangements are made).
- Smoking adjacent to exterior intake air vents.
- Operating or storing unnecessary mechanical equipment in the building; however, necessary mechanical equipment may not be able to be removed, in which case it should be documented in the field notes.
- Allowing unnecessary containers of gasoline or oil to remain within the building. Heating fuel tanks inside buildings should be vented outside the building, or their vents should be temporarily sealed to prevent off-gassing inside the building.
- Cleaning, waxing, or polishing furniture, floors, or other woodwork with petroleum- or oil-based products.
- Using air fresheners or odor eliminators.
- Engaging in work tasks that use materials containing volatile chemicals.

- Building maintenance activities involving products containing volatile chemicals.
- Lawn mowing.
- Applying pesticides or insecticides.
- Wearing or bringing dry-cleaned clothing into the building.

The list above was based on guidance and may be modified to reflect the conditions of the site.

To collect an indoor air sample for VOC analysis using TO-15 and TO-15 SIM, the flow controller will be connected to the top of the Summa canister using Swagelok connections. A short length (hereafter, "short length" refers to approximately 3 to 4 in.) of tubing may be connected to the top of the flow controller to assist with placement of the sample collection point.

A shut-in test will be performed on the Summa canister and flow regulator and/or sample tubing connections to insure there are no leaks.

Indoor air samples will be collected from the breathing zone by placing the intake end of the sampling tubes approximately 3 to 5 ft above the floor (within the breathing zone), in high-use areas. For the Summa canisters (6-L), the laboratory-set flow rate will result in an 24-hour sample period. To initiate sample collection, the Summa canister valve will be opened to allow air to enter the Summa canister.

Periodically throughout the 24-hour sampling period for the Summa canisters, the pressure inside the canister will be monitored and the time required to reach the desired end sampling pressure (approximately -5 in. Hg) will be recorded and included on the chain-of-custody record. To terminate sample collection, the Summa canister valve will be closed and the sample will be contained within the canister. The sample identification and collection date/time will be recorded on the sample label attached to each Summa canister. To verify the final pressure following sample collection, the vacuum of the Summa canister can be measured using the same handheld pressure gauge that was used to verify the initial pressure. The initial and final pressures will be recorded on the chain-of-custody record. Digital photographs will be collected of the indoor air sample setup and sampling train.

A duplicate indoor air would consist of setting two Summa canisters with flow regulators side by side with identical sample start and stop times.

Pressure Differential Monitoring

The pressure differential between indoor air and outdoor air will be determined at each building during each indoor air sampling event, using a micromanometer (e.g., a FLUKE 922 Airflow Meter) as follows:

1. Press "pressure" to enter the pressure mode.

2. Connect a single hose to the input port (Input +), while leaving the reference port (Ref -) unconnected.
3. With the tubing open to ambient conditions (inside the structure), press and hold the zero button to calibrate the meter.

Place the input hose outside, while leaving the meter inside. The micromanometer will display the differential pressure outdoors with respect to the pressure indoors. For instance, a positive reading means that the air outside is positively pressured with respect to the air inside.

4. OUTDOOR AMBIENT AIR SAMPLING PROCEDURES

In addition to the indoor air samples, outdoor ambient air samples will be collected. Ambient air samples will be collected during the same 24-hour period as the indoor air samples, which represent outdoor air conditions for the entire sampling area. The ambient air samples will be collected in a laboratory batch-certified Summa canister, regulated for a 24-hour sample collection. A section of Teflon or Teflon-lined tubing that is identified as laboratory- or food-grade quality will be extended from the Summa canister to collect the ambient air sample from the breathing zone at approximately 3 to 5 ft above ground surface. Outdoor ambient air samples will be collected at a minimum of one (1) per day during the indoor air monitoring program. Based on scheduling and overall distribution of indoor air sampling locations, it will determine if more than one (1) ambient air sample is needed per day.

The following procedures for the collection of outdoor ambient air samples are based on applicable guidance and practical experience during the implementation of field activities on other projects. The procedures are intended to provide the specific process to follow to collect outdoor air samples. However, the procedures are also intended to be flexible enough to implement based on conditions encountered in the field.

Outdoor ambient air samples will be collected from outside of each building retained for vapor intrusion assessment from an area generally upwind of the building.

The procedures to collect an outdoor ambient air sample are identical to those described for indoor air samples. The Summa canisters will require temporary shelter during the 24-hour sample collection process, and should be secured from potential tampering. A 1- to 2-ft length of sample tubing may be attached to the canister to provide a drip loop, so that moisture will not directly enter the sampling orifice. Adhesives or materials potentially containing volatile constituents should be avoided when securing the outdoor air sampling apparatuses. Digital photographs will be collected of the outdoor air sample setup and sampling location.

Appendix B

Field Forms

DAILY FIELD ACTIVITIES SUMMARY REPORT			
PROJECT NAME: Petro-Chemical Systems, Inc. (Turtle Bayou) Remedial Action			
Date:	Shift Beginning: hours		Shift Ending: hours
RAC II Contract No.: EP-W-06-004			Task Order No.: 0134-RARA-0681
EPA Region 6 TOM: Raji Josiam			Project Manager: April Ballweg
Site Manager:			Data Manager:
Project Geologist:			SHSO:
Personnel on site	**Name**	**Affiliation**	**Reason for being on site**
EPA:			
EA:			
DBS&A:			
Subcontractors:			
Other:			
Work Performed			
Report prepared by (name and date)			

Groundwater Collection Field Form

Well ID: _____ **Sample ID:** _____ **Sample Time:** _____

Casing diameter/type: | Well location: | Weather:

Screened interval(s): | Sampling personnel:

Total depth: | Sampling method:

Initial depth to water (w/o pump): | Water level indicator:

Final depth to water (w/o pump): | Water quality meter: YSI

Measuring point: **North side of casing** | Pump depth setting: | Pump type/model:

	Δ < 1° C	Δ < 3%	Δ <10%	Δ <10%	Δ < 0.1 pH	Δ < 10 mV	Δ < 0.3 ft	< 1L/min	Δ < 10 NTU		Parameter Stabilization Limits (3 consecutive readings)
Time	**Temp (°C)**	**Conductivity (mS/cm)** or **(µS/cm)**	**DO (%)**	**DO (mg/L)**	**pH**	**ORP (mV)**	**Water Level (feet btoc)**	**Flow Rate (L/min)** or **(mL/min)**	**Turbidity (NTU)**	**Purge Volume (L)** or **(mL)**	**Additional Comments**

Note:

Parameter Stabilization Limits:
(3 consecutive readings) for percent difference type parameters
Percent difference formula =
ABS[((first reading - second reading)/first reading) x 100]
Ex: Readings 12, 16, 15, 13
((12-16)/12)*100 = 33% ((16-15)/16)*100 = 6%
((15-13)/15)*100 = 13% In example, stabilization has not occurred.

Analyses: (circle those collected and indicate the number of containers)			
VOCs	Metals	Pesticides	Dioxins and Furans
SVOCS	Hexavalent Chromium	Aroclors	Tetraethyl Lead

Recorded By:_____

Date: _____

HydraSleeve™ Sampler Groundwater Sampling Data Sheet

Well ID: _____ **Sample ID:** _____ **Sample Time:** _____

Well owner/location/residence: _____

Street address: _____ Sampling personnel: _____

Weather: _____

HYDRASLEEVE™ SAMPLER DEPTH (feet)	SAMPLE TIME	NOTES/COMMENTS

Field Parameters:

Time:						
Specific Conductance:						µS/cm or mS/cm
pH:						
Temperature:						ºF or ºC
ORP:						mV
Turbidity:						NTU

Notes/Comments: _____

Recorded By: _____

BUILDING INVENTORY AND INDOOR AIR SAMPLING QUESTIONNAIRE

This form should be prepared by a person familiar with indoor air assessments with assistance from a person knowledgeable about the building. Complete this form for each building where interior samples (e.g., indoor air, crawlspace, or sub-slab soil gas samples) will be collected. Section I of this form should be used to assist in selecting an investigative strategy during work plan development. Section II should be used to assist in identifying complicating factors during a pre-sampling building walkthrough.

Preparer's Name_____ Date/Time Prepared_____

Preparer's Affiliation_____ Phone No._____

Purpose of Investigation_____

SECTION I: BUILDING INVENTORY

1. OCCUPANT OR BUILDING PERSONNEL:

Interviewed: Y / N

Last Name_____ First Name_____

Address_____

City_____

Phone No._____

Number of Occupants/people at this location_____ Age of Occupants_____

2. OWNER or LANDLORD: (Check if same as occupant_____.)

Interviewed: Y / N

Last Name_____ First Name_____

Address_____

City_____

Phone No._____

3. BUILDING CHARACTERISTICS

Type of Building: (Circle appropriate response.)

Residential	School	Commercial/Multi-use
Industrial	Church	Other_____

If the property is residential, what type? (Circle appropriate response.)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouse/Condo
Modular	Log Home	Other_____

If multiple units, how many?_____

If the property is commercial, what type?

Business types(s)_____

Does it include residences (i.e., multi-use)? Y / N If yes, how many? _____

Other characteristics:

Number of floors_____ Building age_____

Is the building insulated? Y / N How airtight? Tight / Average / Not Tight

Have occupants noticed chemical odors in the building? Y / N

If yes, please describe:_____

_____

4. AIRFLOW

Use air current tubes, tracer smoke, or knowledge about the building to evaluate airflow patterns and qualitatively describe:

Airflow between floors

_____

_____

_____

Airflow in building near suspected source

_____

_____

_____

Outdoor air infiltration

_____

_____

_____

Infiltration into air ducts

_____

_____

_____

5. BASEMENT AND CONSTRUCTION CHARACTERISTICS (Circle all that apply.)

a. Above-grade construction:	wood frame	log	concrete	brick
	constructed on pilings with enclosed air space		constructed on pilings with open air space	
b. Basement type:	full	crawlspace	slab-on-grade	other_____
c. Basement floor:	concrete	dirt	stone	other _____
d. Basement floor:	unsealed	sealed	sealed with_____	
e. Foundation walls:	poured	block	stone	other _____
f. Foundation walls:	unsealed	sealed	sealed with _____	
g. The basement is:	wet	damp	dry	
h. The basement is:	finished	unfinished	partially finished	
i. Sump present?	Y / N			
j. Water in sump?	Y / N / not applicable			

Basement or lowest level depth below grade_____(feet).

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, and drains).

_____

_____

6. HEATING, VENTING, AND AIR CONDITIONING (Circle all that apply.)

Type of heating system(s) used in this building: (Circle all that apply – not just primary.)

Hot air circulation	Heat pump	Hot water baseboard	
Space heaters	Stream radiation	Radiant floor	
Electric baseboard	Wood stove	Outdoor wood boiler	Other_____

The primary type of fuel used is:

Natural gas	Fuel oil	Kerosene
Electric	Propane	Solar
Wood	Coal	

Domestic hot water tank is fueled by:_____

Boiler/furnace is located in: Basement Outdoors Main floor Other_____

Do any of the heating appliances have cold-air intakes? Y / N

Type of air conditioning or ventilation used in this building:

Central air	Window units	Open windows	None
Commercial HVAC	Heat-recovery system	Passive air system	

Are there air distribution ducts present? Y / N

Describe the ventilation system in the building, its condition where visible, and the tightness of duct joints. Indicate the location of air supply and exhaust points on the floor plan.

_____

_____

_____

_____

Is there a radon mitigation system for the building/structure? Y / N Date of Installation _____

Is the system active or passive? Active/Passive

7. OCCUPANCY

Is basement/lowest level occupied? Full-time Occasionally Seldom Almost never

Level	**General Use of Each Floor (e.g., family room, bedroom, laundry, workshop, or storage).**
Basement	
1st Floor	
2nd Floor	
3rd Floor	

8. WATER AND SEWAGE

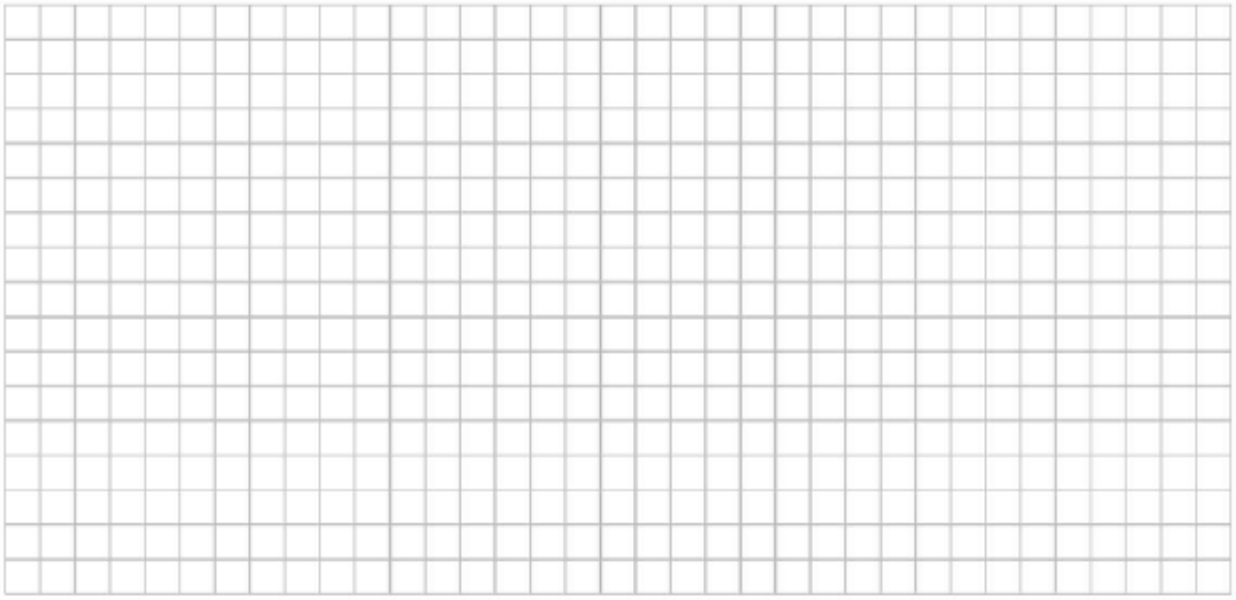
Water supply: Public water Drilled well Driven well Dug well Other_____

Sewage disposal: Public sewer Septic tank Leach field Dry well Other_____

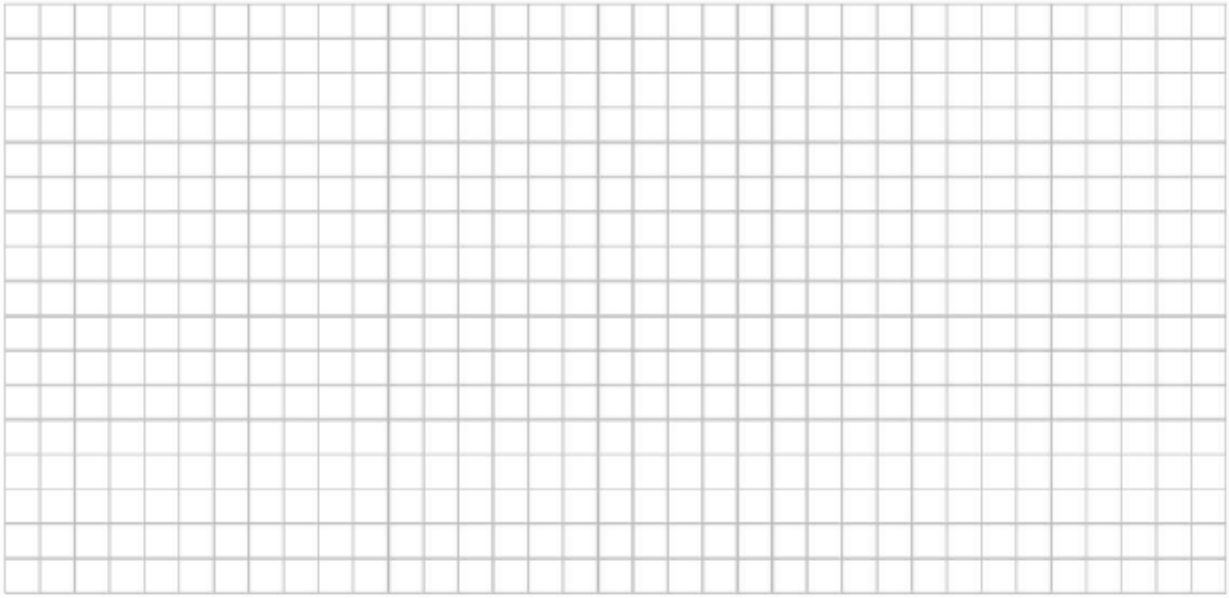
9. FLOOR PLANS

Draw a plan view sketch of the basement and first floor of the building. Indicate air sampling locations, possible indoor air pollution sources and photoionization detector (PID) meter readings. If the building does not have a basement, please note that.

Basement:



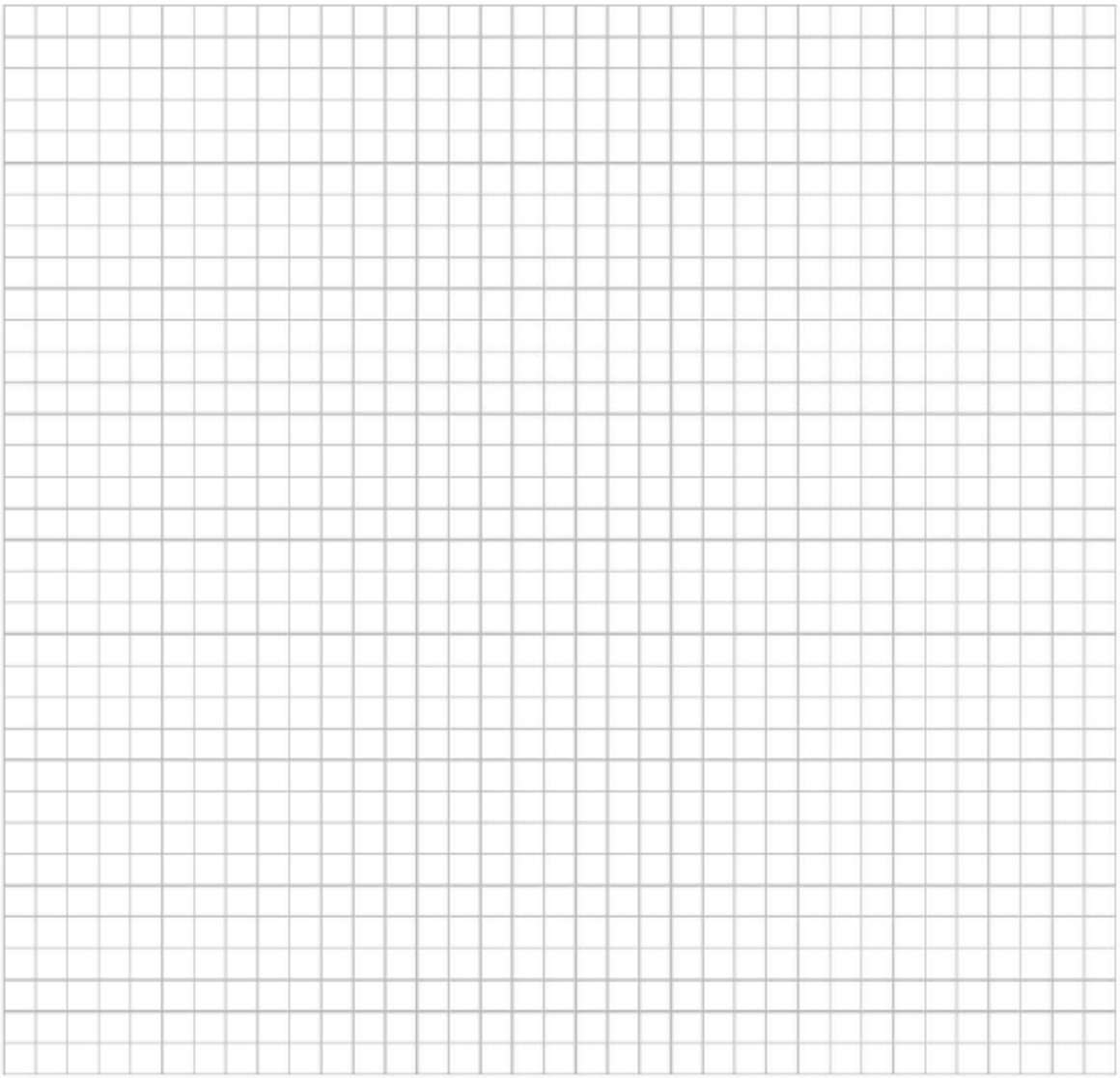
First Floor:



10. OUTDOOR PLOT

Draw a sketch of the area surrounding the building being sampled. If applicable, provide information on spill locations, potential air contamination sources (e.g., industries, gas stations, repair shops, landfills, etc.), outdoor air sampling locations, and PID meter readings.

Also indicate compass direction, wind direction, and speed during sampling; the location of the well and septic system, if applicable; and a qualifying statement to help locate the site on a topographic map.



SECTION II: INDOOR AIR SAMPLING QUESTIONNAIRE

This section should be completed during a pre-sampling walk-through. If indoor air sources of COPCs are identified and removed, consider ventilating the building prior to sampling. However, ventilation and heating systems should be operating normally for 24 hours prior to sampling.

a) 1. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

Is there an attached garage? Y / N

Does the garage have a separate heating unit? Y / N / NA

Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, ATV, or car) Y / N /NA

Please specify_____

Has the building ever had a fire? Y / N When?_____

Is a kerosene or unvented gas space heater present? Y / N Where?_____

Is there a workshop or hobby/craft area? Y / N Where and type_____

Is there smoking in the building? Y / N How frequently?_____

Has painting/staining been done in the last 6 months? Y / N Where and when?_____

Is there new carpet, drapes or other textiles? Y / N Where and when?_____

Is there a kitchen exhaust fan? Y / N If yes, where is it vented?_____

Is there a bathroom exhaust fan? Y / N If yes, where is it vented?_____

Is there a clothes dryer? Y / N If yes, is it vented outside? Y / N

Are cleaning products, cosmetic products, or pesticides used that could interfere with indoor air sampling? Y / N

If yes, please describe_____

_____

_____

Do any of the building occupants use solvents at work? Y / N

(For example, is the building used for chemical manufacturing or a laboratory, auto mechanic or auto body shop, painting shop, fuel oil delivery area, or do any of the occupants work as a boiler mechanic, pesticide applicator, or cosmetologist?)

If yes, what types of solvents are used?_____

If yes, are his/her/their clothes washed at work? Y / N

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, use dry cleaning regularly (weekly) No

Yes, use dry cleaning infrequently (monthly or less) Unknown

Yes, work at a dry cleaning services

2. PRODUCT INVENTORY FORM (For use during building walk-through.)

Make and model of field instrument used:_____

List specific products found in the residence that have the potential to affect indoor air quality:

Location	Product Description	Site (units)	Condition[1]	Chemical Ingredients	Field Instrument Reading (units)	Photo[2] Y / N

[1] Describe the condition of the product containers as **Unopened (UO), Used (U),** or **Deteriorated (D)**.

[2] Photographs of the front and back of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

This form was modified from:

ITRC (Interstate Technology and Regulatory Council). 2007. *Vapor Intrusion Pathway: A Practical Guideline*. VI-1. Washington, D.C.: Interstate Technology and Regulatory Council, Vapor Intrusion Team. Available at: www.itrcweb.org.

FIELD SAMPLING FORM FOR VAPOR INTRUSION ASSESSMENT

Samplers:	Site ID / Bldg ID	**EA Project #:** 14342134 **Client:** EPA Region 6 **Site:** Petro-Chemical Systems, Inc., Liberty, TX **Description:** Vapor Intrusion Assessment Sampling

Location ID: _____

Sample Type: ☐ Indoor Air ☐ Sub-Slab ☐ Outdoor

Air Duplicate: ☐ Yes ☐ No

Probe Installation Date/Time: _____

Slab Thickness:_____Probe Length: _____

Helium Leak Check Date/Time: _____

He% Shroud_____ He% Tedlar Bag_____ VOC Purge _____

Shut In Check PSI drop in 1 minute: _____

TO-15 SIM

Summa Sample ID: _____

Summa Canister ID: _____

Initial Canister Gauge Pressure: _____

Flow Control ID: _____

Flow Control Rate: _____

Canister Start Time/Date: _____

Canister End Time/Date: _____

Final Canister Gauge Pressure: _____

Comments/Observations:

_____

_____

_____

_____

_____

_____

Appendix C

SOM02.4 Organic Target Analyte List and CRQLs

EPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

ORGANIC SUPERFUND METHODS

Multi-Media, Multi-Concentration

SOM02.4
October 2016

STATEMENT OF WORK

TABLE OF CONTENTS

ORGANIC ABBREVIATIONS/ACRONYM LIST	
ABBREVIATION/ACRONYM	DEFINITION
ASB	Analytical Services Branch
ASB CLP COR	Analytical Services Branch Contract Laboratory Program Contracting Officer's Representative
ASE	Accelerated Solvent Extractor
BFB	4-bromofluorobenzene
BNA	Base Neutral Acid
%Breakdown	Percent Breakdown
°C	Degrees Celsius (unit of measurement)
CAS	Chemical Abstracts Service
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CF	Calibration Factor
CFR	Code of Federal Regulations
CLP	EPA Contract Laboratory Program
cm	Centimeter (unit of measurement)
CO	Contracting Officer
COC	Chain of Custody
COR	Contracting Officer's Representative
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
%D	Percent Difference
DF	Dilution Factor
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DRD	Data Receipt Date
DTD	Document Type Definition
EDD	Electronic Data Deliverable
EI	Electron Ionization
EICP	Extracted Ion Current Profile
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
g	Gram (unit of measurement)
GC	Gas Chromatography
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
HPLC	High Performance Liquid Chromatography
HRS	Hazard Ranking System
ICAL	Initial Calibration
ICV	Initial Calibration Verification
ID	Identifier
IPC	Instrument Performance Check
IR	Infrared
IUPAC	International Union of Pure and Applied Chemistry
K-D	Kuderna-Danish
L	Liter (unit of measurement)
Lab	Laboratory
lb	Pound (unit of measurement)
LCS	Laboratory Control Sample
LRD	Laboratory Receipt Date

ORGANIC ABBREVIATIONS/ACRONYM LIST	
ABBREVIATION/ACRONYM	DEFINITION
MA	Modified Analysis
MDL	Method Detection Limits
mg	Milligram (unit of measurement)
mL	Milliliter (unit of measurement)
mm	Millimeter (unit of measurement)
MS	Mass Spectrometry
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MTBE	Methyl tert-butyl ether
µL	Microliter (unit of measurement)
µm	Micrometer (unit of measurement)
NCS	Non-Client Sample
ng	Nanogram (unit of measurement)
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
OSRTI	EPA Office of Superfund Remediation and Technology Innovation
PAH	Polynuclear Aromatic Hydrocarbon
PCP	Pentachlorophenol
PDF	Portable Document Format
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
PFE	Pressurized Fluid Extraction
PFK	Perfluorokerosene
PRPs	Potentially Responsible Parties
Psi	Pounds Per Square Inch (unit of measurement)
P/T	Purge-and-trap
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
QMP	Quality Management Plan
%R	Percent Recovery
RESC	Resolution Check Standard
RIC	Reconstructed Ion Chromatogram
RPM	Revolutions Per Minute (unit of measurement)
RRF	Relative Response Factor
RRT	Relative Retention Time
%RSD	Percent Relative Standard Deviation
RPD	Relative Percent Difference
RT	Retention Time
%S	Percent Solids
SA	Spike Added
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	Standard Deviation
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SICP	Selected Ion Current Profile
SIM	Selected Ion Monitoring
SMO	Sample Management Office

ORGANIC ABBREVIATIONS/ACRONYM LIST	
ABBREVIATION/ACRONYM	DEFINITION
SOP	Standard Operating Procedure
SOW	Statement of Work
SPLP	Synthetic Precipitation Leaching Procedure
SVOA	Semivolatile Organic Analyte
TAL	Target Analyte List
TBA	Tetrabutylammonium
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TR	Traffic Report
TR/COC	Traffic Report/Chain of Custody
UTF-8	Unicode Transformation Format - 8 bit
UV	Ultraviolet
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compound
VTSR	Validated Time of Sample Receipt
W3C	World Wide Web Consortium
XML	eXtensible Markup Language
ZHE	Zero Headspace Extraction

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EXHIBIT A

SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

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1.0 PURPOSE

The purpose of this analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (EPA), in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other EPA Program Offices, as well as customers outside the Agency, that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

This Statement of Work (SOW) provides a contractual framework for laboratories to perform analytical services. This framework applies EPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 51 Trace Volatiles, 51 Low/Medium Volatiles, 69 Semivolatiles, 18 Semivolatiles by SIM, 21 Pesticides, and 9 Aroclors in aqueous/water and soil/sediment samples. The SOW also includes Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) leachate extraction procedures. The analytical service contract provides the methods to be used and the specific contractual requirements by which the EPA will evaluate the data.

3.0 DATA USES

This analytical service provides data used for a variety of purposes, such as: determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate clean-up actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including site inspections, Hazard Ranking System (HRS) scoring, remedial investigation/feasibility studies, remedial design, treatability studies, and removal actions.

In addition, the Contractor must be aware of the importance of maintaining the integrity of data generated under the contract, since it is used to make major decisions regarding public health and environmental welfare. The data may also be used in litigation against Potentially Responsible Parties (PRPs) in the enforcement of Superfund legislation.

4.0 SUMMARY OF REQUIREMENTS

The SOW is comprised of eight exhibits:

- Exhibit A – Summary of Requirements
- Exhibit B – Reporting and Deliverables Requirements
- Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits
- Exhibit D – Analytical Methods
- Exhibit E – Quality Systems
- Exhibit F - Programmatic Quality Assurance/Quality Control Elements
- Exhibit G – Glossary of Terms
- Exhibit H – Format for Electronic Data Deliverables

4.1 Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced.

4.1.1 Sample Receiving, Storage, and Disposal

The Contractor will receive samples from potential hazardous waste sites and shall store and maintain these samples under proper chain of custody (COC) procedures. The Contractor shall follow procedures outlined in Section 5.0 of this Exhibit for proper sample receipt and handling as well as each Exhibit D – Analytical Methods for proper storage and disposal of unused portion of samples. All anomalies and identified issues shall be communicated to the EPA via the CLP Sample Management Office (SMO) Contractor.

4.1.2 Sample Preparation and Analysis

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.1.2.1 The Contractor shall prepare samples as described in the respective Exhibit D – Analytical Methods for the requested analysis type. Sample preparation methods shall remain consistent for all samples analyzed within a Sample Delivery Group (SDG).

4.1.3 Sample Reporting and Resubmission of Data

4.1.3.1 Required formats for the reporting of data are found in Exhibit B – Reporting and Deliverables Requirements and Exhibit H – Format for Electronic Data Deliverables. The Contractor shall be responsible for completing and submitting analysis data sheets and electronic data as requested in a format specified in this SOW and within the time specified in Exhibit B – Reporting and Deliverables Requirements, Section 1.1.

4.1.3.2 Use of formats other than those approved will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format will be required at no additional cost to the Government.

4.1.4 Quality Assurance/Quality Control

The Contractor shall maintain a Quality Assurance Project Plan (QAPP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, and all documentation required during data collection, as well as the Quality Assurance (QA) measures performed by management to ensure acceptable data production.

4.1.4.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D – Analytical Methods and F – Programmatic Quality Assurance/Quality Control Elements. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit E – Quality Systems, and shall be reported in accordance with Exhibit B – Reporting and Deliverables Requirements and Exhibit H – Format for Electronic Data Deliverables.

4.1.4.2 Additional QC shall be conducted in the form of the analysis of Performance Evaluation (PE) samples submitted to the laboratory by the EPA. Unacceptable results of all such QC or PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to the EPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by the EPA, as well as meeting the contract requirements for analysis (Exhibit D – Analytical Methods); QA/QC (Exhibit F – Programmatic Quality Assurance/Quality Control Elements); data reporting and other deliverables (Exhibits B – Reporting and Deliverables Requirements and H – Format for Electronic Data Deliverables); and sample custody, sample documentation, and Standard Operating Procedure (SOP) documentation (Exhibit E – Quality Systems). As an alternative to data rejection, the EPA may require reanalysis of noncompliant samples. Reanalysis will be performed by the Contractor at no additional cost to the EPA.

4.1.5 Modified Analysis

The Contractor may be requested by the EPA to perform a Modified Analysis (MA). The modifications may include, but are not limited to: modified preparation or analysis procedures; additional analytes; sample matrices other than those present in the SOW; and/or lower quantitation limits. The requests will be made in writing, prior to sample scheduling. All contract requirements specified in the SOW/Specifications will remain in effect unless specifically modified.

5.0 SAMPLE RECEIPT AND HANDLING

5.1 Chain of Custody

The Contractor shall receive and maintain samples under proper COC procedures. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete SDG File (CSF) (See Exhibit B – Reporting and Deliverables Requirements). The Contractor shall establish and use appropriate procedures to handle confidential information received from the EPA.

5.2 Sample Scheduling

5.2.1 Sample shipments to the Contractor’s facility will be scheduled and coordinated by the CLP SMO. The EPA may request analyses that include all or a subset of the Target Analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits. The EPA may also request modified analyses due to the nature of the samples or project requirements. The Contractor shall communicate with SMO personnel as necessary, throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

5.2.2 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation defined in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

5.3 Sample Shipments

5.3.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including weekends.

5.3.2 Unless otherwise instructed by the EPA Region or originating sampler, the Contractor shall be required to routinely return sample shipping containers to the appropriate sampling office within 14 calendar days following shipment receipt. This shipment must be done via ground transportation only pending receipt of a valid return authorization, unless specifically instructed to do otherwise. The Contractor will be provided a shipping mechanism by the EPA Region or originating sampler (e.g., field sampler). The Contractor shall ensure that the account numbers provided are used only for the return of Government-owned shipping containers.

5.3.2.1 The Contractor shall remove packing and other materials from the shipping containers before each pick-up and shall ensure that the shipping containers are clean. The Contractor can determine from visual inspection whether the shipping container is clean.

5.4 Sample Receipt

5.4.1 If insufficient sample amount (less than the required amount) is received to perform the analyses, the Contractor shall notify SMO and proceed with the analysis of the sample at reduced volume. The Contractor shall document this issue in the SDG Narrative.

5.4.2 If the Contractor receives broken sample containers, with enough remaining sample to perform sample analysis, but potentially not enough volume to analyze any possible re-extractions/reanalyses, the Contractor shall note the issue in the SDG Narrative, proceed with analysis of the samples, and notify SMO. If re-extraction/ reanalyses are necessary, the Contractor shall contact SMO. The Contractor shall document the provided resolution in the SDG Narrative.

5.4.3 If the Contractor encounters other problems with samples or related documentation [e.g., mixed media, sample pH, sample documentation and paperwork such as Traffic Report/Chain of Custody (TR/COC) Records not with shipment, sample and TR/COC Record do not correspond], the Contractor shall immediately contact SMO for resolution.

5.4.4 Shipping Container Temperature Monitoring

5.4.4.1 To monitor the temperature of the sample shipping container more effectively, a sample shipping container temperature indicator bottle may be included with each shipping container shipped. The applicable temperature blank will be clearly labeled.

5.4.4.2 When a shipping container temperature indicator bottle is included in the sample shipping container, the Contractor shall use the supplied shipping container temperature indicator bottle to determine the shipping container temperature. The temperature of the sample shipping container shall be measured and recorded immediately upon opening the shipping container, and prior to unpacking the samples or removing the packing material.

5.4.4.3 To determine the temperature of the shipping container, the Contractor shall locate the shipping container temperature indicator bottle in the sample shipping container, invert it several times, remove the cap, and insert a calibrated [National Institute of Standards and Technology (NIST)-traceable] thermometer into the shipping container temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the thermometer used shall be capable of measuring and registering the temperature of the shipping container with an accuracy of ±1°C.

5.4.4.4 If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining shipping container temperature. Other devices (e.g., infrared thermometer) which can measure temperature may be used if they can be calibrated to ±1°C.

5.4.4.5 If a temperature indicator bottle is not present in the shipping container, and the temperature of the shipping container is not less than or equal to 6°C, the Contractor shall note the issue, and the method used to determine the temperature, in the SDG Narrative and proceed with analysis of the samples. If the temperature exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the EPA for instructions on how to proceed. SMO will in turn notify the Contractor of the EPA's decision. The Contractor shall document the EPA's decision and the EPA Sample Numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

5.4.4.6 Liquid bearing thermometers such as mercury or alcohol thermometers shall be traceable to NIST calibration and verified at least annually, and whenever the thermometer has been exposed to temperature extremes. The correction factor shall be indicated on the thermometer, and the date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection. The NIST thermometer shall be recalibrated at least every five years or whenever the thermometer has been exposed to temperature extremes.

Digital thermometers, thermocouples, and other similar electronic temperature measuring devices shall be calibrated at least quarterly. The date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection.

When an infrared (IR) detection device is used to measure the temperature of samples, the device shall be verified at least every six months using an NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient (20-30°C), iced (4°C), and frozen (0 to -5°C). Each day of use, a single check of the IR shall be made by measuring the temperature of a bottle of water, that contains a calibrated thermometer, at the temperature of interest. Agreement between the two readings should be within 0.5°C, or the device shall be recalibrated. The daily checks of the IR shall be documented and the records maintained on file.

5.4.5 Recording Sample pH

5.4.5.1 The pH for all aqueous/water samples received by the Contractor shall be measured, using a method capable of demonstrating that proper preservation was performed (e.g., pH test strips, electronic hand-held pen, pH meter), and recorded. The pH shall be determined using a small aliquot of the sample to prevent contamination. Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

5.4.5.2 All pens and pH meter electrodes shall be rinsed with reagent water between sample readings.

5.5 Sample Case

Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case Number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

5.5.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or
- Each 20 samples (excluding PE samples) within a Case; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with receipt of the first sample in the SDG).
- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining an SDG.
- All samples scheduled with the same level of deliverables.

5.5.2 Samples may be assigned to SDGs by matrix (i.e., all soil/sediment in one SDG, all aqueous/water in another), at the discretion of the laboratory. If PE samples are received within a Case, they shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received and shall <u>not</u> be made retroactively. The SDG may exceed the 20 samples limit since the limitation excludes PE samples.

5.5.3 Each sample received by the Contractor will be labeled with an EPA Sample Number and accompanied by a TR/COC Record bearing the Sample Number and descriptive information regarding the sample. The EPA Sample Numbers are continuous, without spaces or hyphens. If the sample numbers do not conform to this requirement, contact SMO. The Contractor shall complete and sign the TR/COC Record, recording the date of sample receipt and sample condition on receipt for each sample container.

5.5.3.1 The Contractor shall follow the instructions given on the TR/COC Record in choosing the QC samples, when such information is provided. If no QC sample is designated on the TR/COC Record, the Contractor shall select a sample and notify SMO for EPA Regional acceptance. SMO shall contact the EPA Region for confirmation immediately after notification.

5.5.3.2 If the Sampler designated two (or more) samples as QC for the same matrix, and the QC samples are not specifically labeled with the analysis they are to be used for, then the Contractor is to contact SMO to report the issue. SMO shall then contact the EPA Region and notify the Contractor of the EPA Regional decision. If the Sampler did not designate QC samples, then the Contractor is to select a sample for QC and to contact SMO to report the issue.

5.5.4 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. 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 TR/COC Record.

5.5.5 The Contractor shall submit electronic copy(ies) of signed TR/COC Record as Portable Document Format (PDF) file(s) for all samples in an SDG to SMO via the Superfund Analytical Services SMO Portal at https://epasmoweb.fedcsc.com within 3 working days following the receipt of the last sample in the SDG. TR/COC Records shall be submitted with their SDG information as specified in Exhibit B – Reporting and Deliverables Requirements.

5.5.6 The EPA Case Numbers, SDG Numbers, and EPA Sample Numbers shall be used by the Contractor in identifying samples received under this contract, both verbally and in reports/correspondence.

5.5.7 The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

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EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B – Reporting and Deliverables Requirements

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table identifies the contract reporting and deliverables requirements, and specifies the distribution that is required for each deliverable.

TABLE 1. DELIVERABLE SCHEDULE

Item		No. of Copies[1]	Delivery Schedule	Distribution		
				SMO	Region	QATS
A.	Sample Traffic Reports/Chain of Custody (TR/COC) Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).	X		
B.[2,3]	Complete SDG File (CSF)	1	XX[4] days after Validated Time of Sample Receipt (VTSR) of last sample in SDG.		X	
C.[2,5,7]	Copy of CSF and Hardcopy Data in Portable Document Format (PDF) Format	1	XX[4] days after VTSR of last sample in SDG.	X		
D.[2,6]	Preliminary Results (Volatiles Analyses)	1	Within 48 hours after receipt of each sample at laboratory, if requested.	X	X	
	Preliminary Results (Semivolatiles, Pesticides, and Aroclor Analyses)	1	Within 72 hours after receipt of each sample at laboratory, if requested.	X	X	
E.[2,7]	Electronic Data Deliverable (EDD)	1	XX[4] days after VTSR of last sample in SDG.	X		
F.[2]	Proficiency Testing (PT) Audits	1	XX[4] days after VTSR of last sample in SDG.	X		

TABLE 1. DELIVERABLE SCHEDULE (CON'T)

Item		No. of Copies[1]	Delivery Schedule	Distribution		
				SMO	Region	QATS
G.[7]	Determination of Method Detection Limits (MDL)	1	MDL values in spreadsheet format specified in Appendix A of Exhibit H prior to analysis of field samples, annually thereafter, and after major instrument adjustments to SMO and QATS. MDL study data prior to analysis of field samples, annually thereafter, and after major instrument adjustments to QATS only. Submission of all deliverables within 7 days of determinations.	X		X
H.	Standard Operating Procedures (SOPs)	1	Submit within 60 days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 4.0) Submit amended documents within 14 days of amended SOP(s) as directed in Exhibit E, Section 4.4.			X
I.	Quality Assurance Project Plan (QAPP)	1	Submit within XX[4] days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 3.0) Submit amended documents within 14 days of amended QAPP as directed in Exhibit E, Section 3.3.			X

TABLE 1. DELIVERABLE SCHEDULE (CON'T)

Item		No. of Copies[1]	Delivery Schedule	Distribution		
				SMO	Region	QATS
J.	Instrument Electronic Data	Lot	Retain for 3 years after data submission of the reconciled CSF. Submit within 7 days of receipt of written request, to recipients as directed. (See Exhibit F, Section 8.3)	As Directed		X
K.	Extracts	Lot	Retain for 1 year after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		
L.	Samples	Lot	Retain for 60 days after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		

Footnotes:

[1] The number of copies specified is the number of copies required to be delivered to each recipient.

[2] **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE.** Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date that all samples have been delivered. **If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.**

[3] CSF will contain the original Sample Data for Level 2a, 2b, and 3 deliverables, plus all of the original documents described in Exhibit B, Section 2.4.

[4] The number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.

[5] Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the U.S. Environmental Protection Agency's Regional Contract Laboratory Program Contracting Officer's Representative(EPA Regional CLP COR) and Analytical Services Branch CLP COR (ASB CLP COR). Supplemental data (i.e., logbooks) may be requested in writing from the EPA Regional staff or the ASB CLP COR. All written communication sent by the EPA must include the EPA Regional CLP COR in the distribution list. If the EPA Regional CLP COR has not been included in the distribution list, contact the ASB CLP COR.

[6] If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form 1-OR sample analyses and field Quality Control (QC) analyses. The Contractor shall provide the SMO copy via the EPA Electronic Data Exchange and Evaluation System (EXES) at https://epasmoweb.fedcsc.com as a PDF file as preliminary results. The PDF file name should be PR_Case Number_SDG Number_Contract Number_Method. Sample TR/COC Records and SDG Cover Page (per Exhibit B, Section 2.7.1) shall be submitted with the Preliminary Results. The designated Regional recipient shall receive the Preliminary Results as a PDF file or in alternative electronic formats (e.g., Microsoft® Word) via email. The Contractor will be notified of the email address and format at the time of sample scheduling.

NOTE: Preliminary Results Delivery Schedule:

If a sample requiring Preliminary Results arrives at the laboratory before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received at the laboratory after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

[7] The Contractor shall provide SMO the electronic files via EXES at https://epasmoweb.fedcsc.com.

1.2 Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1 – Deliverable Schedule.

Sample Management Office (SMO)[1]:

Delivery instructions shall be provided upon contract award.

EPA Region:

SMO will provide the Contractor with the list of addressees for data delivery for the 10 EPA Regions. SMO will provide the Contractor with updated EPA Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

EPA Regional CLP Contracting Officer's Representative:

SMO will provide the Contractor with the list of addresses for the EPA Regional CLP CORs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

Quality Assurance Technical Support (QATS)[2]:

Delivery instructions shall be provided upon contract award.

[1] SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by the EPA.
[2] QATS is a Contractor-operated facility operating under the QATS contract awarded and administered by the EPA.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1 (for hardcopy) and Exhibit H (for electronic). The required content and form of each deliverable are described in this Exhibit. All reports and documentation **shall be**:

- Legible;
- Clearly labeled and completed in accordance with instructions in this Exhibit;
- Arranged in the order specified in this Exhibit;
- Paginated sequentially according to instructions in this Exhibit; and
- Double-sided.
- Information reported on the forms listed in this Exhibit [excluding the Sample Log-In Sheet (DC-1) and the Complete SDG File (CSF) Inventory Sheet (DC-2)] must be computer-generated.
- The Contractor shall use EPA Case Numbers, SDG Numbers, and EPA Sample Numbers to identify samples received under this contract, verbally, electronically, and in reports and correspondence. The Contract Number and the Statement of Work (SOW) Number shall be specified in all correspondence. The Modification Analysis Number (MA No.) shall also be included for all Modified Analyses.

2.1.1 The Contractor shall submit Staged Electronic Data Deliverable (SEDD) Level 2a, Level 2b, or Level 3 deliverables as specified at the time of scheduling.

- Level 2a deliverables consist of a specified limited subset of the data reporting forms as specified in this Exhibit.
- Level 2b deliverables include all data reporting forms as specified in this Exhibit.
- Level 3 deliverables include all data reporting forms and supporting raw data as specified in this Exhibit.

2.1.2 Section 3.0 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with all required data. Section 4.0 of this Exhibit contains the required Data Reporting Forms in Agency-specified format. Data elements and instructions for electronically reporting data are contained in Exhibit H – Format for Electronic Data Deliverables.

2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the EPA.

2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through an EPA Regional CLP COR action, or through an EPA Regional data reviewer’s request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients (SMO and EPA Region) and to the EPA’s designated recipient when a written request for a copy of

the CSF has been made within 5 business days (3 business days for a 7-day turnaround) of receipt of the request. A cover letter shall be included which describes what data are being delivered, to which EPA Case Number(s) and SDG Number(s) the data pertains, and who requested the data. Corrected data submitted as "Additional Data" at the request of an EPA Regional data reviewer shall only include the affected pages and be accompanied by a revised SDG Narrative (described in Section 2.4.5 of this Exhibit) documenting the reason(s) for the resubmittal.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to both contractual data recipients (SMO and EPA Region), and to the EPA's designated recipient when a written request for a copy of the CSF has been made, within 6 business days of receipt of the request. In all instances, the Contractor shall include a cover sheet (Laboratory Response to Results of Contract Compliance Screening). Electronic deliverables shall be submitted or resubmitted to SMO only. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the EPA Region.

2.3 Sample Traffic Report/Chain of Custody Records

2.3.1 Each sample received by the Contractor shall be labeled with an EPA Sample Number and will be accompanied by a TR/COC Record bearing the Sample Number and descriptive information regarding the sample. The Contractor shall complete the TR/COC Record, recording the date of sample receipt, verifying the number of samples, and signing the TR/COC Record.

2.3.1.1 Upon receipt, the Contractor shall sign for the receipt of samples in the COC Record section. The laboratory Sample Custodian or designated recipient opening and verifying the contents of the shipping container shall then verify receipt of all samples identified within the CLP Traffic Report section and sign and date the signature box located in the CLP Traffic Report section. If a non-CLP TR/COC Record is submitted with the samples (e.g., a Regional TR/COC Record), then the Contractor shall: (1) record the receipt date of the samples and sign the TR/COC Record to maintain the chain-of-custody, and (2) the Sample Custodian or designated recipient shall sign and date the TR/COC Record to verify sample information.

NOTE: If the laboratory is requested to transfer samples to another facility, the Contractor shall date and enter the name of the facility to where the samples will be transferred on the CLP TR/COC Record and document in the SDG Narrative.

2.3.1.2 The Contractor shall also enter the SDG Number, Case Number, and the Laboratory Contract Number on the CLP TR/COC Record. The EPA Sample Number of the first sample received in the SDG is the SDG Number. When several samples are received together in the first SDG shipment, the SDG Number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. Under no circumstances should any SDG Number be replicated within a Case. If necessary, select an alternative sample number for the SDG Number. The SDG Number is also reported on all data reporting forms (see Exhibit B, Section 3.0 - Form Instructions).

2.3.2 The Contractor shall submit TR/COC Records in SDG sets (i.e., TR/COC Records for all samples in an SDG), with an SDG Definition Sheet attached. The SDG Definition Sheet shall contain the following items:

- Laboratory Name;
- Contract Number;
- Modified Analysis Number (if applicable);
- Case Number;
- List of the method/analysis for each sample; and
- List of EPA Sample Numbers of all samples in the SDG, identifying the first and last samples received, and their Laboratory Receipt Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations).

2.3.3 EPA Sample Numbers are continuous, without spaces or hyphens. The original Sample TR/COC Record page, with laboratory receipt information and signed with an original Contractor signature, shall be submitted for each sample in the SDG.

2.3.4 If samples are received at the laboratory with multi-sample TR/COC Records, all the samples on one multi-sample TR/COC Record may not necessarily be in the same SDG. In this instance, the Contractor must make the appropriate number of photocopies of the TR/COC Record and submit one copy with each SDG Definition Sheet.

2.4 Complete Sample Delivery Group File

The CSF is described in this section. Sections 2.4.7 through 2.4.11 are specific to the individual analytical methods. If analysis by one or more of the analytical methods is not required, then those method sections are not required as a deliverable. Each method section shall include data for analysis of all samples in one SDG, including field samples, calibrations, QC samples, and supporting documentation. The CSF shall be complete before submission. The CSF shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package).

2.4.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to the EPA with another Case/SDG. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3.0 and 4.0; and organized according to Form DC-2.

NOTE: The Contractor shall retain a legible electronic (PDF) or hardcopy of the CSF for 365 days after submission of the reconciled data package to the Government. After this time, the Contractor may dispose of the package.

2.4.2 The CSF shall consist of the following original documents:

- Completed SDG Cover Page with signature and date
- EPA Sample TR/COC Record
- Completed and signed Sample Log-In Sheet [Form DC-1]
- Completed and signed Full Organics Complete SDG File (CSF) Inventory Sheet [Form DC-2]
- SDG Narrative
- All original shipping documents, including, but not limited to, the following documents:
  - Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);
  - Sample Tags (if present) sealed in plastic bags; and
  - All original receiving documents, including, but not limited to, other receiving forms or copies of receiving logbooks.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to the EPA, as well as copies that are altered in any fashion, are also deliverables to the EPA. Send the original to the EPA Region and a copy to SMO. Send to the EPA's designated recipient only upon written request.

2.4.3 For Level 3 deliverables, all original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:

- Percent Solids Log;
- Original preparation, cleanup, and analysis forms, or copies of preparation, cleanup, and analysis logbook pages;
- Internal sample and sample extract transfer Chain of Custody Records;
- Screening records;
- All instrument output, including strip charts, Gel Permeation Chromatography (GPC), High Performance Liquid Chromatography (HPLC), and all cleanup activities; and
- Performance Evaluation (PE) Instruction forms.

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

- Communication logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

If the Contractor does submit SDG-specific documents to the EPA after the submission of the CSF, the documents shall be identified with submission codes. For example, if a page or pages were submitted with errors, the corrected pages would be identified with

the Case and SDG Number, and the code R#, where the "#" is incremented for any subsequent resubmissions. If a page has been left out of a CSF, it must be submitted with the code A#. If the entire CSF is to be resubmitted, it must be designated with the code RS#. A revised Form DC-2 should be submitted, and the submission codes and locations of the documents in the CSF shall be recorded in the "Other Records" section on the revised Form DC-2.

2.4.5 SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory Name, SOW Number, Contract Number, Case Number, SDG Number, Modified Analysis Number (if applicable), and detailed documentation of any QC, sample, shipment, and/or analytical problems encountered in processing the samples reported in the CSF.

2.4.5.1 The Contractor shall include any technical and administrative problems encountered, and the resolution or corrective actions taken. These problems may include, but are not limited to interference problems encountered during analysis, dilutions, reanalyses and/or re-extractions performed, and any problems with the analysis of samples.

2.4.5.2 Document the alternative temperature technique used, if applicable, to determine shipping container temperature if a temperature indicator bottle is not present in the shipping container.

2.4.5.3 The Contractor shall also provide at least one example of each type of calculation, including relative response factors or calibration factors (CFs), as well as sample results to allow the recalculation of sample results from raw instrument output.

2.4.5.4 The Contractor shall also include a discussion of any SOW Modified Analyses. This includes attaching a copy of the approved modification form to the SDG Narrative.

2.4.5.5 The Contractor shall also identify and explain any differences which exist between the Form(s) 1-OR and supporting documentation provided in the data package and those previously provided as Preliminary Results.

2.4.5.6 SDG Narrative associated attachments, including, but not limited to:

- Gas Chromatography (GC) column information; and
- Unequivocal cross reference of laboratory to EPA Sample Numbers.

2.4.5.7 When submitting corrected data as "Additional Data" at the request of an EPA Regional data reviewer, the Contractor shall include a revised SDG Narrative documenting the reason(s) for the resubmittal.

2.4.6 SDG Cover Page

Cover Page for the organic analyses data shall include: Laboratory Name; Laboratory Code; Contract Number; Case Number; SDG Number; Modified Analysis Number (MA No.) (if appropriate); SOW Number; EPA Sample Numbers in alphanumeric order cross-referenced with Laboratory Sample ID numbers; and Analytical Method.

2.4.6.1 The SDG Cover Page shall contain 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 in the

SDG Narrative. Release of the data contained in this hardcopy Complete SDG File and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as 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.

2.4.7 Trace Volatile Organics Sample Data Forms and Raw Data

2.4.7.1 Quality Control Summary

2.4.7.1.1 Deuterated Monitoring Compound Recovery [Form 2A-OR and Form 2B-OR].

2.4.7.1.2 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]. This data shall be provided upon the EPA Region's request for analysis of Matrix Spike/Matrix Spike Duplicates (MS/MSDs).

2.4.7.1.3 Method Blank Summary [Form 4-OR]. If more than a single form is necessary, forms shall be in chronological order by date of analysis of the blank, by instrument.

2.4.7.1.4 GC/MS Instrument Performance Check [Form 5-OR]. If more than a single form is necessary, forms shall be in chronological order, by instrument. Not required for Level 2a deliverables.

2.4.7.1.5 Internal Standard Area and Retention Time Summary [Form 8A-OR]. If more than a single form is necessary, forms shall be arranged in chronological order, by instrument. Not required for Level 2a deliverables.

2.4.7.2 Sample Data

Sample data shall be submitted with the organic analysis data reporting forms for all samples in the SDG. Data shall be arranged in increasing alphanumeric EPA Sample Number order. For Level 3 deliverables, the forms for each sample analysis shall be followed by the sample raw data for that analysis.

2.4.7.2.1 Organic Analysis Data Sheet [Form 1A-OR and Form 1B-OR]. Tabulated analytical results (identification and quantitation) of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. 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(s) in the SDG Narrative.

2.4.7.2.2 Appropriate concentration units shall be specified and entered on Form 1A-OR. The quantitative values shall be reported in units of micrograms/Liter (µg/L) for aqueous/water samples (no other units are acceptable). Analytical results shall be reported to two significant figures.

2.4.7.2.3 Tentatively Identified Compounds (TICs) [Form 1B-OR]. Form 1B-OR is the tabulated list of the highest probable match for up to 30 organic compounds that are not trace volatile, low/medium volatile, or semivolatile target analytes, Deuterated Monitoring Compounds (DMCs), internal standard compounds, or alkanes, and are not listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The

tabulated list includes the Chemical Abstracts Service (CAS) Number (if applicable), tentative identification, and estimated concentration. This form shall be included, even if no compounds are found. No duplicated CAS numbers should be reported for TICs. Follow the instructions in Exhibit D – Trace Concentrations of Volatile Organic Compounds Analysis, Section 11.1.2.4 when reporting TICs.

2.4.7.2.4 Reconstructed Total Ion Chromatograms (for each sample including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest non-solvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- Gas Chromatograph/Mass Spectrometer (GC/MS) instrument identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.7.2.4.1 Internal standards and DMCs shall be labeled with the names of analytes, either directly out from the peak or on a printout of Retention Times (RTs) if RTs are printed over the peak. Labeling of other analytes is not required and should not detract from the legibility of the required labels.

2.4.7.2.4.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target analytes, the complete data system report shall be included in the Level 3 CSF, in addition to the reconstructed ion chromatogram. The complete data system report shall include the following information:

- EPA Sample Number;
- Date and time of analysis;
- RT or scan number of identified target analytes;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On-column concentration/amount, including units;
- GC/MS instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.7.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the Extracted Ion Current Profiles (EICPs) of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy

printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all trace volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.7.2.4.4 Other Required Information for Level 3 reporting. For each sample, by each analyte identified, the following items shall be included in the data package:

- Copies of raw spectra and copies of background-subtracted mass spectra of trace volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits that are identified in the sample and corresponding background-subtracted target analyte standard mass spectra. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Analyte names shall be clearly marked on all spectra; and
- Copies of mass spectra of organic analytes not listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits with associated best-match spectra (maximum of three best matches). Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Analyte names shall be clearly marked on all spectra.

2.4.7.3 Standards Data

2.4.7.3.1 GC/MS Initial Calibration Data [Form 6A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the five-point initial calibration, labeled as in Section 2.4.7.2.4. Spectra are not required.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed or for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- Labels for standards shall reflect the concentrations of the non-ketone analytes in µg/L. (If the non-ketone analytes have a concentration of 5.0 µg/L, then the reported label shall be RRF5.0).
- EICPs displaying each manual integration and the corresponding original system integration.

2.4.7.3.2 Initial Calibration Verification and Continuing Calibration Verification for GC/MS [Form 7A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial calibration verifications and all continuing (12-hour) calibration verifications, labeled as in Section 2.4.7.2.4. Spectra are not required.
- When more than one Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV) is performed, forms shall be in chronological order, by instrument. The ICV forms shall be placed together prior to all CCV forms.
- EICPs displaying each manual integration and the corresponding original system integration.

2.4.7.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all trace volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.7.4 Quality Control Data – Raw data only required for Level 3 deliverables.

2.4.7.4.1 4-bromofluorobenzene data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.4.7.2.4.
- Mass listing, labeled as in Section 2.4.7.2.4.
- Reconstructed total ion chromatogram, labeled as in Section 2.4.7.2.4.

2.4.7.4.2 Blank data shall be arranged by type of blank (method, storage, or instrument) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results [Form 1A-OR].
- Tentatively Identified Compounds [Form 1B-OR] even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.7.2.4.
- Target analyte spectra with laboratory-generated standard, labeled as in Section 2.4.7.2.4. 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.

- GC/MS library search spectra for TICs, labeled as in Section 2.4.7.2.4.

- Quantitation/calculation of TIC concentrations.

2.4.7.4.3 Matrix Spike and Matrix Spike Duplicate Data

- Tabulated results [Form 1A-OR] of target analytes is required if MS/MSD analysis is requested at the time of scheduling by the EPA Region. Form 1B-OR is not required.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.7.2.4. Spectra are not required.

2.4.8 Low/Medium Volatile Organics Sample Data Forms and Raw Data

2.4.8.1 Quality Control Summary

2.4.8.1.1 Deuterated Monitoring Compound Recovery [Form 2A-OR and Form 2B-OR]

2.4.8.1.2 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]. This data shall be provided upon the EPA Region's request for analysis of MS/MSDs.

2.4.8.1.3 Method Blank Summary [Form 4-OR]. If more than a single form is necessary, forms shall be in chronological order by date of analysis of the blank, by instrument.

2.4.8.1.4 GC/MS Instrument Performance Check [Form 5-OR]. If more than a single form is necessary, forms shall be in chronological order, by instrument. Not required for Level 2a deliverables.

2.4.8.1.5 Internal Standard Area and Retention Time Summary [Form 8A-OR]. If more than a single form is necessary, forms shall be in chronological order, by instrument. Not required for Level 2a deliverables.

2.4.8.2 Sample Data

Sample data shall be submitted with the organic analysis data reporting forms for all samples in the SDG. Data shall be arranged in increasing alphanumeric EPA Sample Number order. For Level 3 deliverables, the forms for each sample analysis shall be followed by the sample raw data for that analysis.

2.4.8.2.1 Organic Analysis Data Sheet [Form 1A-OR and Form 1B-OR]. Tabulated analytical results (identification and quantitation) of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. 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(s) in the SDG Narrative.

2.4.8.2.2 Appropriate concentration units shall be specified and entered on Form 1A-OR. The quantitative values shall be reported in units of µg/L for aqueous/water samples, milligrams/Liter (mg/L) for Toxicity Characteristic Leaching Procedure (TCLP) leachate samples, and micrograms/kilogram (µg/kg) for soil/sediment samples (no other units are acceptable). Results for soil/sediment samples shall be reported on a dry

weight basis. Analytical results shall be reported to two significant figures.

2.4.8.2.3 Tentatively Identified Compounds (TICs) [Form 1B-OR]. Form 1B-OR is the tabulated list of the highest probable match for up to 30 organic compounds that are not trace volatile, low/medium volatile, or semivolatile target analytes, DMCs, internal standard compounds, or alkanes, and are not listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The tabulated list includes the CAS Number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. No duplicated CAS numbers should be reported for TICs. Follow the instructions in Exhibit D – Low/Medium Concentrations of Volatile Organic Compounds Analysis, Section 11.1.2.4 when reporting TICs.

2.4.8.2.4 Reconstructed Total Ion Chromatograms (for each sample including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest non-solvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- GC/MS instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.8.2.4.1 Internal standards and DMCs shall be labeled with the names of analytes, either directly out from the peak or on a printout of RTs if RTs are printed over the peak. Labeling of other analytes is not required and should not detract from the legibility of the required labels.

2.4.8.2.4.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target analytes, the complete data system report shall be included in the Level 3 CSF, in addition to the reconstructed ion chromatogram. The complete data system report shall include the following information:

- EPA Sample Number;
- Date and time of analysis;
- RT or scan number of identified target analytes;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On-column concentration/amount, including units;
- GC/MS instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.8.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all low/medium volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.8.2.4.4 Other Required Information for Level 3 reporting. For each sample, by each analyte identified, the following items shall be included in the data package:

- Copies of raw spectra and copies of background-subtracted mass spectra of low/medium volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits that are identified in the sample and corresponding background-subtracted target analyte standard mass spectra. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Analyte names shall be clearly marked on all spectra; and

- Copies of mass spectra of organic compounds not listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits with associated best-match spectra (maximum of three best matches). Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Analyte names shall be clearly marked on all spectra.

2.4.8.3 Standards Data

2.4.8.3.1 GC/MS Initial Calibration Data [Form 6A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the five-point initial calibration, labeled as in Section 2.4.8.2.4. Spectra are not required.

- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed or for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.

- Labels for standards shall reflect the concentrations of the non-ketone analytes in µg/L. (If the non-ketone analytes have a concentration of 5.0 µg/L, then the reported label shall be RRF5.0).

NOTE: For low-level soil sediment samples, the concentration of the low standard is 2.5 µg/L. Since 10 milliliter (mL) purge volumes are required for low-level soil standards, the reported label shall be RRF2.5.

- EICPs displaying each manual integration and the corresponding original system integration.

2.4.8.3.2 Initial Calibration Verification and Continuing Calibration Verification for GC/MS [Form 7A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial calibration verifications and all continuing (12-hour) calibration verifications, labeled as in Section 2.4.8.2.4. Spectra are not required.
- When more than one ICV or CCV is performed, forms shall be in chronological order, by instrument. The ICV forms shall be placed together prior to all CCV forms.
- EICPs displaying each manual integration and the corresponding original system integration.

2.4.8.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all low/medium volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.8.4 Quality Control Data – Raw data only required for Level 3 deliverables.

2.4.8.4.1 4-bromofluorobenzene (BFB) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.4.8.2.4.
- Mass listing, labeled as in Section 2.4.8.2.4.
- Reconstructed total ion chromatogram, labeled as in Section 2.4.8.2.4.

2.4.8.4.2 Blank data shall be arranged by type of blank (method, storage, and instrument) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results [Form 1A-OR].

- Tentatively Identified Compounds [Form 1B-OR] even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.8.2.4.
- Target analyte spectra with laboratory-generated standard, labeled as in Section 2.4.8.2.4. Data systems that are incapable of dual display shall provide spectra in the following order:
  - Raw target analyte spectra.
  - Enhanced or background-subtracted spectra.
  - Laboratory-generated standard spectra.
- GC/MS library search spectra for TICs, labeled as in Section 2.4.8.2.4.
- Quantitation/calculation of TIC concentrations.

2.4.8.4.3 Matrix Spike and Matrix Spike Duplicate Data

- Tabulated results [Form 1A-OR] of target analytes are required if MS/MSD analysis is requested at the time of scheduling by the EPA Region. Form 1B-OR is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.8.2.4. Spectra are not required.

2.4.9 Semivolatile Organics Sample Data Forms and Raw Data

2.4.9.1 Quality Control Summary

2.4.9.1.1 Deuterated Monitoring Compound Recovery [Form 2A-OR and Form 2B-OR]

2.4.9.1.2 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]. This data shall be provided upon the EPA Region's request for analysis of MS/MSDs.

2.4.9.1.3 Method Blank Summary [Form 4-OR]. If more than a single form is necessary, forms shall be in chronological order by date of analysis of the blank, by instrument.

2.4.9.1.4 GC/MS Instrument Performance Check [Form 5-OR]. If more than a single form is necessary, forms shall be in chronological order, by instrument. Not required for Level 2a deliverables.

NOTE: For the Selected Ion Monitoring (SIM) analysis technique, this form is required for analytical sequence although Instrument Performance Check information on this form is optional.

2.4.9.1.5 Internal Standard Area and Retention Time Summary [Form 8A-OR]. If more than a single form is necessary, forms shall be in chronological order, by instrument. Not required for Level 2a deliverables.

2.4.9.2 Sample Data

Sample data shall be submitted with the organic analysis data reporting forms for all samples in the SDG. Data shall be arranged in increasing alphanumeric EPA Sample Number order. For Level 3 deliverables, the forms for each sample analysis shall be followed by the sample raw data for that analysis.

Semivolatile sample data for SIM analysis shall be arranged together with the rest of the SIM Semivolatiles data at the end of the subsection.

2.4.9.2.1 Organic Analysis Data Sheet [Form 1A-OR and Form 1B-OR]. Tabulated analytical results (identification and quantitation) of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. 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(s) in the SDG Narrative.

2.4.9.2.2 Appropriate concentration units shall be specified and entered on Form 1A-OR. The quantitative values shall be reported in units of µg/L for aqueous/water samples, mg/L for TCLP leachate samples, and µg/kg for soil/sediment samples (no other units are acceptable). Results for soil/sediment samples shall be reported on a dry weight basis. Analytical results shall be reported to two significant figures.

2.4.9.2.3 Tentatively Identified Compounds (TICs) [Form 1B-OR]. Form 1B-OR is the tabulated list of the highest probable match for up to 30 organic compounds that are not trace volatile, low/medium volatile, and semivolatile target analytes, DMCs, internal standard compounds, or alkanes, and are not listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The tabulated list includes the CAS Number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. No duplicated CAS numbers should be reported for TICs. Follow the instructions in Exhibit D – Semivolatiles Organic Compounds Analysis Section 11.1.2.5 when reporting TICs.

2.4.9.2.4 Reconstructed Total Ion Chromatograms (for each sample including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest non-solvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- GC/MS instrument and column identifier;
- Laboratory File Identifier; and
- 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.

2.4.9.2.4.1 Internal standards and DMCs shall be labeled with the names of analytes, either directly out from the peak or on a printout of RTs if RTs are printed over the peak. Labeling of other analytes is not required and should not detract from the legibility of the required labels.

2.4.9.2.4.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target analytes, the complete data system report shall be included in the Level 3 CSF, in addition to the reconstructed ion chromatogram. The complete data system report shall include the following information:

- EPA Sample Number;
- Date and time of analysis;
- RT or scan number of identified target analytes;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On-column concentration/amount, including units;
- GC/MS instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.9.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all semivolatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.9.2.4.4 Other Required Information for Level 3 reporting. For each sample, by each analyte identified, the following items shall be included in the data package:

- Copies of raw spectra and copies of background-subtracted mass spectra of semivolatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits that are identified in the sample and corresponding background-subtracted target analyte standard mass spectra. This includes target analytes that are identified during the optional analysis using the SIM technique. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Analyte names shall be clearly marked on all spectra; and
- Copies of mass spectra of organic analytes not listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits with associated best-match spectra (maximum of three best matches). Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS

instrument identifier. Analyte names shall be clearly marked on all spectra.

2.4.9.3 Standards Data

2.4.9.3.1 GC/MS Initial Calibration Data [Form 6A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the five-point initial calibration, labeled as in Section 2.4.9.2.4. Spectra are not required.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed or for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- Labels for standards shall reflect the concentrations of the analytes in ng/µL. (If the target analytes have a concentration of 5.0 ng/µL, then the reported label shall be RRF5.0).
- EICPs displaying each manual integration and the corresponding original system integration.

2.4.9.3.2 Initial Calibration Verification and Continuing Calibration Verification for GC/MS [Form 7A-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contactor shall submit the following raw data:

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial calibration verifications and all continuing (12-hour) calibration verifications, labeled as in Section 2.4.9.2.4. Spectra are not required.
- When more than one ICV or CCV is performed, forms shall be in chronological order, by instrument. The ICV forms shall be placed together prior to all CCV forms.
- EICPs displaying each manual integration and the corresponding original system integration.

2.4.9.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. The hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all semivolatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

2.4.9.4 Quality Control Data – Raw data only required for Level 3 deliverables.

2.4.9.4.1 Decafluorotriphenylphosphine (DFTPP) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.4.9.2.4.
- Mass listing, labeled as in Section 2.4.9.2.4.
- Reconstructed total ion chromatogram, labeled as in Section 2.4.9.2.4.

2.4.9.4.2 Blank data shall be arranged by type of blank (method) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results [Form 1A-OR].
- Tentatively Identified Compounds [Form 1B-OR] even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.9.2.4.
- Target analyte spectra with laboratory-generated standard, labeled as in Section 2.4.9.2.4. Data systems that are incapable of dual display shall provide spectra in the following order:
  - Raw target analyte spectra.
  - Enhanced or background-subtracted spectra.
  - Laboratory-generated standard spectra.
- GC/MS library search spectra for TICs, labeled as in Section 2.4.9.2.4.
- Quantitation/calculation of TIC concentrations.

2.4.9.4.3 Matrix Spike and Matrix Spike Duplicate Data

- Tabulated results [Form 1A-OR] of target analytes are required if MS/MSD analysis is requested at the time of scheduling by the EPA Region. Form 1B-OR is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.4.9.2.4. Spectra are not required.

2.4.10 Pesticide Organics Sample Data Forms and Raw Data

2.4.10.1 Quality Control Summary

2.4.10.1.1 Surrogate Recovery [Form 2C-OR]

2.4.10.1.2 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]. MS/MSD analysis is required for the pesticide method unless otherwise specified by the EPA Region. See Exhibit D – Pesticides Analysis for frequency.

2.4.10.1.3 Laboratory Control Sample Recovery [Form 3B-OR]

2.4.10.1.4 Method Blank Summary [Form 4-OR]. If more than a single form is necessary, forms shall be in chronological order by date of analysis of the blank, by instrument.

2.4.10.2 Sample Data

Sample data shall be submitted with the organic analysis data reporting forms for all samples in the SDG. Data shall be arranged in increasing alphanumeric EPA Sample Number order. For Level 3 deliverables, the form for each sample shall be followed by the sample raw data for both analyses.

2.4.10.2.1 Organic Analysis Data Sheet [Form 1A-OR]. The lower concentration of the requested analytes tabulated (identification and quantitation) using both analytical GC columns must be reported. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. 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(s) in the SDG Narrative.

2.4.10.2.2 Appropriate concentration units shall be specified and entered on Form 1A-OR. The quantitative values shall be reported in units of µg/L for aqueous/water samples, mg/L for TCLP leachate samples, and µg/kg for soil/sediment samples (no other units are acceptable). Results for soil/sediment samples shall be reported on a dry weight basis. Analytical results shall be reported to two significant figures.

2.4.10.2.3 Chromatograms (for each sample including dilutions and reanalyses). Chromatograms shall be normalized to the largest non-solvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- Gas Chromatograph/Electron Capture Detector (GC/ECD) instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.10.2.4 Surrogates shall be labeled with the names of analytes, either directly out from the peak or on a printout of RTs if RTs are printed over the peak. Labeling of other analytes is not required and should not detract from the legibility of the required labels.

2.4.10.2.4.1 If automated data system procedures are used for preliminary identification and/or quantitation of the target analytes, the complete data system report shall be included in the Level 3 CSF, in addition to the chromatogram. The complete data system report shall include the following information:

- EPA Sample Number;
- Date and time of analysis;
- RT of identified target analytes;
- Peak area responses used for quantitation;
- On-column concentration/amount, including units;
- GC/ECD instrument and column identifier;

- Laboratory File Identifier; and
- Analyst ID.

2.4.10.2.4.2 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all pesticide target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits and surrogates.

2.4.10.2.4.3 Other Required Information for Level 3 reporting. For each sample, by each analyte identified, the following items shall be included in the data package:

- Copies of raw chromatograms from both GC columns used to analyze the pesticide target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. Chromatograms shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/ECD instrument identifier. Analyte names shall be clearly marked on all chromatograms.

2.4.10.3 Standards Data

2.4.10.3.1 Initial Calibration Data [Form 6B-OR, 6C-OR, 6D-OR, 6E-OR, 6F-OR, and 6G-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Pesticide standard(s) chromatograms and quantitation reports for the five-point initial calibration, labeled as in Section 2.4.10.2.3. The CFs and RTs for each concentration level of Pesticide target analytes and surrogates.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- Labels for standards shall reflect the concentration levels of the initial calibration standards. The lowest level is labeled as CF1, the next level is labeled sequentially as CF2, and the 5th level is labeled as CF5.
- Chromatograms displaying each manual integration and the corresponding original system integration.

2.4.10.3.2 Continuing Calibration Verification Data [Form 7B-OR, 7C-OR, and 7D-OR] shall be included in order by instrument, for each instrument used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Pesticide standard(s) chromatograms and quantitation reports for all continuing (12-hour) calibration verifications, labeled as in Section 2.4.10.2.3.
- When more than one CCV is performed, forms shall be in chronological order, by instrument.
- Chromatograms displaying each manual integration and the corresponding original system integration.

2.4.10.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all pesticide target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits and surrogates.

2.4.10.3.4 Analytical Sequence [Form 8B-OR] for pesticide analyses must be included for each GC column used. Not required for Level 2a deliverables.

2.4.10.3.5 Florisil Cartridge Check [Form 9A-OR] for mandatory cleanup of sample extracts. Florisil check chromatograms and quantitation reports for each lot of Florisil cartridge used to cleanup up sample extracts must be included. Not required for Level 2a deliverables.

2.4.10.3.6 GPC Calibration Verification [Form 9B-OR] for sample extracts that underwent GPC cleanup. GPC calibration verification and GPC blank chromatograms and quantitation reports must be reported weekly for each GPC system used to cleanup sample extracts included in the SDG. Not required for Level 2a deliverables.

2.4.10.3.7 Identification Summary [Form 10A-OR and 10B-OR] for all samples with positively identified single and multi-component analytes, in order by increasing EPA Sample Number. Form 10B-OR not required for Level 2a deliverables.

2.4.10.4 Quality Control Data – Raw data only required for Level 3 deliverables.

2.4.10.4.1 QC data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/ECD system utilized.

- Chromatogram, labeled as in Section 2.4.10.2.3.

2.4.10.4.2 Blank data shall be arranged by type of blank (method and instrument) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results [Form 1A-OR].
- Chromatograms and quantitation report from each analytical GC column used for analysis, labeled as in Section 2.4.10.2.3.
- Quantitation/calculation of analyte and surrogate concentrations.

2.4.10.4.3 Laboratory Control Sample Data

- Tabulated results [Form 1A-OR] of target analytes from each analytical column used for analysis.
- Chromatogram(s) and quantitation report(s), labeled as in Section 2.4.10.2.3.

2.4.10.4.4 Matrix Spike and Matrix Spike Duplicate Data

- Tabulated results [Form 1A-OR] of target analytes from each analytical column used for analysis.
- Chromatogram(s) and quantitation report(s), labeled as in Section 2.4.10.2.3.

2.4.11 Aroclor Organics Sample Data Forms and Raw Data

2.4.11.1 Quality Control Summary

2.4.11.1.1 Surrogate Recovery [Form 2C-OR]

2.4.11.1.2 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]. MS/MSD analysis is required for the Aroclor method unless otherwise specified by the EPA Region. See Exhibit D – Aroclors Analysis for frequency.

2.4.11.1.3 Laboratory Control Sample Recovery [Form 3B-OR]

2.4.11.1.4 Method Blank Summary [Form 4-OR]. If more than a single form is necessary, forms shall be in chronological order by date of analysis of the blank, by instrument.

2.4.11.2 Sample Data

Sample data shall be submitted with the organic analysis data reporting forms for all samples in the SDG. Data shall be arranged in increasing alphanumeric EPA Sample Number order. For Level 3 deliverables, the form for each sample shall be followed by the sample raw data for both analyses.

2.4.11.2.1 Organic Analysis Data Sheet [Form 1A-OR]. The lower concentration of the requested analytes tabulated (identification and quantitation) using both analytical GC columns must be reported. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. 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(s) in the SDG Narrative.

2.4.11.2.2 Appropriate concentration units shall be specified and entered on Form 1A-OR. The quantitative values shall be reported in units of µg/L for aqueous/water samples and µg/kg for soil/sediment samples (no other units are acceptable). Results for soil/sediment samples shall be reported on a dry

weight basis. Analytical results shall be reported to two significant figures.

2.4.11.2.3 Chromatograms (for each sample including dilutions and reanalyses). Chromatograms shall be normalized to the largest non-solvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- GC/ECD instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.11.2.4 Surrogates shall be labeled with the names of analytes, either directly out from the peak or on a printout of RTs if RTs are printed over the peak. Labeling of other analytes is not required and should not detract from the legibility of the required labels.

2.4.11.2.4.1 If automated data system procedures are used for preliminary identification and/or quantitation of the target analytes, the complete data system report shall be included in Level 3 CSF, in addition to the chromatogram. The complete data system report shall include the following information:

- EPA Sample Number;
- Date and time of analysis;
- RT of identified target analytes;
- Peak area responses used for quantitation;
- On-column concentration/amount, including units;
- GC/ECD instrument and column identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.4.11.2.4.2 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all Aroclor target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits and surrogates.

2.4.11.2.4.3 Other Required Information for Level 3 reporting. For each sample, by each analyte identified, the following items shall be included in the data package:

- Copies of raw chromatograms from both GC columns used to analyze the Aroclor target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. Chromatograms shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/ECD instrument identifier. Analyte names shall be clearly marked on all chromatograms.

2.4.11.3 Standards Data

2.4.11.3.1 Initial Calibration Data [Form 6D-OR, 6E-OR, and 6F-OR] shall be included in order by instrument, if more than one instrument is used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Aroclor standard(s) chromatograms and quantitation reports for the five-point initial calibration and for the single-point calibration, labeled as in Section 2.4.11.2.3. The CFs and RTs for each concentration level of Aroclor target analytes and surrogates.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed or for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- Labels for standards shall reflect the concentration levels of the initial calibration standards. The lowest level is labeled as CF1, the next level is labeled sequentially as CF2, and the 5th level is labeled as CF5.
- Chromatograms displaying each manual integration and the corresponding original system integration.

2.4.11.3.2 Continuing Calibration Verification Data [Form 7D-OR] shall be included in order by instrument, for each instrument used. Not required for Level 2a deliverables. For Level 3 deliverables, the Contractor shall submit the following raw data:

- Aroclor standard(s) chromatograms and quantitation reports for all continuing (12-hour) calibration verifications, labeled as in Section 2.4.11.2.3.
- When more than one CCV is performed, forms shall be in chronological order, by instrument.
- Chromatograms displaying each manual integration and the corresponding original system integration.

2.4.11.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each

integrated area with the letter "m" on the quantitation report, initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all Aroclor target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits and surrogates.

2.4.11.3.4 Analytical Sequence [Form 8B-OR] for Aroclor analyses must be included for each GC column used. Not required for Level 2a deliverables.

2.4.11.3.5 GPC Calibration Verification [Form 9B-OR] for sample extracts that underwent GPC cleanup. GPC calibration verification and GPC blank chromatograms and quantitation reports must be reported weekly for each GPC system used to cleanup sample extracts included in the SDG. Not required for Level 2a deliverables.

2.4.11.3.6 Identification Summary [10B-OR] for all samples with positively identified Aroclor target analytes, in order by increasing EPA Sample Number. Not required for Level 2a deliverables.

2.4.11.4 Quality Control Data – Raw data only required for Level 3 deliverables.

2.4.11.4.1 QC data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/ECD system utilized.

- Chromatogram, labeled as in Section 2.4.11.2.3.

2.4.11.4.2 Blank data shall be arranged by type of blank (method and instrument) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results [Form 1A-OR].
- Chromatograms and quantitation report from each analytical GC column used for analysis, labeled as in Section 2.4.11.2.3.
- Quantitation/calculation of analyte and surrogate concentrations.

2.4.11.4.3 Laboratory Control Sample Data

- Tabulated results [Form 1A-OR] of target analytes from each analytical column used for analysis.
- Chromatogram(s) and quantitation report(s), labeled as in Section 2.4.11.2.3.

2.4.11.4.4 Aroclor Matrix Spike and Matrix Spike Duplicate Data

- Tabulated results [Form 1A-OR] of target analytes from each analytical column used for analysis.
- Chromatogram(s) and quantitation report(s), labeled as in Section 2.4.11.2.3.

2.5 Copy of Complete Sample Delivery Group File

The laboratory shall provide a copy of the CSF and a PDF file to SMO, as specified in Table 1 – Deliverable Schedule, of this Exhibit.

2.6 Electronic Data Deliverables

The Contractor shall provide the required electronic data deliverable as specified in Table 1 – Deliverable Schedule, of this Exhibit.

2.6.1 Electronic Data Delivery in Staged Electronic Data Deliverable

The Contractor shall provide an EDD in SEDD format for Levels 2a, 2b, and 3. The EDD shall include analytical data for all samples in the SDG, as specified in Exhibit H – Format for Electronic Data Deliverables.

2.6.2 Portable Document Format of Complete Sample Delivery Group File

The Contractor shall provide a complete copy of the CSF, and any additional or reconciled hardcopy deliverables, in a PDF file via EXES at https://epasmoweb.fedcsc.com, and follow the naming convention for the PDF file. The format of the PDF file should be HCD_Case Number_SDG Number_Contract Number_Submission Type.

2.6.2.1 The following identifiers are used based on submission type:

TABLE 2. PDF SUBMISSION IDENTIFIERS

Submission Type	**Identifier**
First Submission	FS
Replacement Submission (if a complete replacement of the first submission PDF is required)	RS
Reconciliation Submission	R# (The # character represents the number of the reconciliation. For example, the first reconciliation submission would be identified as R1.)
Additional Data Submission	A# (The # character represents the number of the additional data submissions. For example, the first additional data submission would be identified as A1.)

2.6.2.1.1 The PDF file shall be organized in accordance with the directions provided in Exhibit B, Section 2.0 of the SOW.

2.6.2.1.2 Organic data shall be bookmarked using a hierarchical bookmark structure (i.e., an overview or "parent" bookmark, and a subordinate or "child" bookmark nested underneath the "parent" bookmark). The required hierarchical structure is shown in Table 3 – Hierarchical Bookmark Structure.

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE

Group Bookmark	Parent Bookmark	Child Bookmark
SDG Cover Page, Sample TR/COC Records, Form DC-1, Form DC-2, and SDG Narrative		
Trace Volatile Organic Data	QC Summary	Deuterated Monitoring Compound Recovery
		Matrix Spike and Matrix Spike Duplicate Sample Recovery
		Method Blank Summary
		Instrument Performance Check
		Internal Standard
	Sample Data	Organic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
		Tentatively Identified Compounds (with supporting raw data)
	Standards Data	Initial Calibration
		Initial Calibration Verification
		Continuing Calibration Verification
	QC Data	GC/MS Raw Data
		GC/MS Performance Check Raw Data
		Blanks
		Matrix Spike and Matrix Spike Duplicate Data
		Preparation Logs
		Standard and Reagent Preparation Logs
		Analysis Logs
Low/Medium Volatile Organic Data	QC Summary	Deuterated Monitoring Compound Recovery
		Matrix Spike and Matrix Spike Duplicate Sample Recovery
		Method Blank Summary
		Instrument Performance Check
		Internal Standard
	Sample Data	Organic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
		Tentatively Identified Compounds (with supporting raw data)
	Standards Data	Initial Calibration
		Initial Calibration Verification
		Continuing Calibration Verification
	QC Data	GC/MS Raw Data
		GC/MS Performance Check Raw Data
		Blanks
		Matrix Spike and Matrix Spike Duplicate Data
		Preparation Logs
		Standard and Reagent Preparation Logs
		TCLP/Synthetic Precipitation Leaching Procedure (SPLP) Logbooks
		Analysis Logs

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE (CON'T)

Group Bookmark	Parent Bookmark	Child Bookmark
Semivolatile Organic Data	QC Summary	Deuterated Monitoring Compound Recovery
		Matrix Spike and Matrix Spike Duplicate Sample Recovery
		Method Blank Summary
		Instrument Performance Check
		Internal Standard
	Sample Data	Organic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
		Tentatively Identified Compounds (with supporting raw data)
	Standards Data	Initial Calibration
		Initial Calibration Verification
		Continuing Calibration Verification
	QC Data	GC/MS Raw Data
		GC/MS Performance Check Raw Data
		Blanks
		Matrix Spike and Matrix Spike Duplicate Data
		Preparation Logs
		Standard and Reagent Preparation Logs
		TCLP/SPLP Logbooks
		Analysis Logs
Pesticide Data	QC Summary	Surrogate Recovery
		Matrix Spike and Matrix Spike Duplicate Sample Recovery
		Laboratory Control Sample Recovery
		Method Blank Summary
	Sample Data	Organic Analysis Data Sheet (with supporting raw data) in increasing alphanumeric EPA Sample Number order
	Standards Data	Resolution Checks
		Instrument Performance Checks
		Initial Calibration
		Continuing Calibration Verification
		Analytical Sequence
		Cleanup Checks
		Analyte Identification Summary
	QC Data	Blanks
		Matrix Spike and Matrix Spike Duplicate Data
		Laboratory Control Sample Data
		Preparation Logs
		TCLP/SPLP Logbooks
		Standard and Reagent Preparation Logs
		Analysis Logs

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE (CON'T)

Group Bookmark	Parent Bookmark	Child Bookmark
Aroclor Data	QC Summary	Surrogate Recovery
		Matrix Spike and Matrix Spike Duplicate Sample Recovery
		Laboratory Control Sample Recovery
		Method Blank Summary
	Sample Data	Organic Analysis Data Sheet (with supporting raw data) in increasing alphanumeric EPA Sample Number order
	Standards Data	Initial Calibration
		Continuing Calibration Verification
		Analytical Sequence
		Cleanup Checks
		Analyte Identification Summary
	QC Data	Blanks
		Matrix Spike and Matrix Spike Duplicate Data
		Laboratory Control Sample Data
		Preparation Logs
		Standard and Reagent Preparation Logs
		Analysis Logs
Receiving Documents, Transfer Records, and Miscellaneous	Additional Documents	Receiving Logbooks
		Internal Sample, Sample Extract, and Transfer Chain-of-Custody Records
		PE/PT Instruction Forms
		Communication Logs

2.7 Preliminary Results

The Form(s) 1-OR 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 Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form(s) 1-OR as "Preliminary Results" under the form title (i.e., under Organic Analysis Data Sheet). The Contractor shall also include a disclaimer on all Form(s) 1-OR stating that the "Data results contained on this Form 1-OR are for screening purposes only, and may not have been validated for CLP criteria". Sample TR/COC Records and SDG Cover Page (per Exhibit B, Section 2.7.1) shall be submitted with the Preliminary Results.

2.7.1 The Contractor shall submit the SDG Cover Page following the specifications in Exhibit B, Sections 2.4.6 and 3.4.1. The SDG Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The SDG Cover Page shall contain the following statement, 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 in the SDG Narrative. Release of the data contained in this hardcopy Data Package has been authorized by the Laboratory Manager or the Manager's designee, as 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.

2.8 Method Detection Limits

The Contractor shall perform and report determination of the MDLs by the method specified in Exhibit D – Analytical Methods for each instrument used under this contract.

The Contractor shall deliver all determined MDLs to SMO and QATS electronically in the format described in Appendix A – Format Characteristics for Method Detection Limit Study Data, of Exhibit H – Format for Electronic Deliverables, according to the delivery schedule specified in Table 1 – Deliverable Schedule, of Exhibit B – Reporting and Deliverables Requirements.

Submission of the study data for the determination of method and instrument parameters, to QATS only, shall include the data used to determine the values reported. The Contractor shall provide MDL raw data including sample, calibration, and QC data and supporting documentation, including, but not limited to: Extraction Logs, Standard and Reagent Preparation Logs, and Analysis Logs, where applicable, to QATS only, according to the delivery schedule specified in Table 1 – Deliverable Schedule, of Exhibit B – Reporting and Deliverables Requirements.

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section contains specific instructions for the completion of all required Organic Data Reporting Forms.

3.2 General Information

Values shall be reported on the hardcopy forms according to the respective form instructions in this section.

3.2.1 The data reporting forms discussed in Exhibit B, Section 3.4, and presented in Exhibit B, Section 4.0, have been designed in conjunction with the electronic data format specified in Exhibit H – Format for Electronic Data Deliverables. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID". See Table 4 – Required Forms for Reporting Level, for a listing of required forms by reporting level.

TABLE 4. REQUIRED FORMS FOR REPORTING LEVEL

Level	Required Forms
SEDD 2a	Forms 1, 2, 3, 4
SEDD 2b	Forms 1-10 (all Forms)
SEDD 3	Forms 1-10 (all Forms)

3.2.2 All characters which appear on the data reporting forms presented in Section 4.0 shall be reproduced by the Contractor when submitting data, and the format of the forms submitted shall provide exactly the same information as that shown in the contract. No information may be added, deleted, or moved from its specified position. The names of various fields and analytes (i.e., "Lab Code", "Extract Volume") shall appear as they are listed in Exhibits B – Reporting and Deliverables Requirements, and Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, of this SOW.

3.2.3 Rounding Rules

For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is greater than or equal to 5, the result is to be rounded up; otherwise the result is rounded down. For example, 0.4365 rounds to 0.44 and 102.4443 rounds to 100. Also see "Rounding Rules" in Exhibit G - Glossary of Terms.

3.2.3.1 Before evaluating a number for being in control or out of control of a certain limit [other than the Contract Required Quantitation Limit (CRQL)], the number evaluated shall be rounded using the above rounding rules to the significance reported for that limit. For example, the control limit for a surrogate Percent Recovery (%R) is 30-150%. Then a calculated %R of 150.46 shall be reported on Form 2C-OR as 150, which is within the control limits of 30-150. On the other hand, a calculated %R of 150.5 shall be reported on Form 2C-OR as 151, which is not within the 30-150 percent control limits.

3.3 Header and General Form Information

Six pieces of information are common to the header section of each data reporting form. These are Lab Name, Contract, Lab Code, Case Number (Case No.), Modified Analysis Number (MA No.), and SDG Number (SDG No.). Except as noted below for MA No., this information shall be entered on every form and shall match on all forms.

3.3.1 "Lab Name" shall be the name chosen by the Contractor to identify the laboratory.

3.3.2 "Contract" is the number of the EPA contract under which the analyses were performed.

3.3.3 "Lab Code" is an alphanumeric abbreviation, assigned by the EPA, to identify the laboratory and aid in data processing. This Lab Code will be assigned by the EPA at the time a contract is awarded and shall not be modified by the Contractor, except at the direction of the EPA Contracting Officer (CO). If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same unless and until the Contractor is directed by the EPA CO to use another EPA-assigned Lab Code.

3.3.4 "Case No." is the SMO-assigned Case Number associated with the sample, and reported on the TR/COC Record or sample shipping paperwork.

3.3.5 "MA No." is the EPA-assigned number for analyses performed for an analytical method under the Modified Analysis clause in Exhibit A - Summary of Requirements. If samples are to be analyzed under the Modified Analysis clause, the Contractor shall list the modification reference number on all forms. If the analyses have no modified requirements, leave the "MA No." field blank.

3.3.6 "SDG No." is the SDG Number.

3.3.7 "EPA SAMPLE NO." appears either in the header section, upper right-hand corner of the form, or as the left column of a table summarizing data from a number of samples.

3.3.7.1 All samples, dilutions, reanalyses, leachates, blanks, matrix spikes, matrix spike duplicates, laboratory control samples, and standards shall be identified with an EPA Sample Number. For samples, an EPA Sample Number is the unique identifying number

given on the TR/COC Record or sample shipping records that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 5 - Codes for Labeling Data, must be used.

TABLE 5. CODES FOR LABELING DATA[1,2,3,4,5,6]

Name	Sample Number
Sample in SDG (TCLP/SPLP Leachate included)	XXXXX
Sample or Laboratory QC Not Part of the SDG[6]	ZZZZZ
Matrix Spike[1]	XXXXXMS
Matrix Spike Duplicate[1]	XXXXXMSD
Re-extracted and reanalyzed Sample	XXXXXRX
Re-extracted and reanalyzed Sample at a dilution	XXXXXRXDL
Reanalyzed (re-injected) Sample	XXXXXRE
Reanalyzed (re-injected) Sample at a dilution	XXXXXREDL
Sample analyzed at a dilution	XXXXXDL
Sample analyzed at a secondary dilution	XXXXXDL2
Sample analyzed at a third dilution	XXXXXDL3
Soil/sediment samples analyzed using the medium level method when the low-level analysis of the same sample is also present	XXXXXME
Instrument Calibration Standards:	
Volatile Instrument Performance Checks	BFB##
Semivolatile Instrument Performance Checks	DFTPP##
Volatile Standard[2]	VSTD***##
Semivolatile Standard[2]	SSTD***##
Volatile Initial Calibration Verification	VICV##
Semivolatile Initial Calibration Verification	SICV##
Pesticides Resolution Check	RESC##
Pesticides Performance Evaluation Mixture	PEM##
Pesticides Individual Mixture A (CS*)[3]	INDA*##
Pesticides Individual Mixture B (CS*)[3]	INDB*##
Pesticides Individual Mixture C (CS*)[3]	INDC*##
Toxaphene (CS*)[3]	TOXAPH*##
Aroclor 1016 (CS*)[3]	AR1016*##
Aroclor 1221 (CS*)[3]	AR1221*##
Aroclor 1232 (CS*)[3]	AR1232*##
Aroclor 1242 (CS*)[3]	AR1242*##
Aroclor 1248 (CS*)[3]	AR1248*##
Aroclor 1254 (CS*)[3]	AR1254*##
Aroclor 1260 (CS*)[3]	AR1260*##
Aroclor 1262 (CS*)[3]	AR1262*##
Aroclor 1268 (CS*)[3]	AR1268*##
Aroclor 1016/1260 Mixture (CS*)[3]	AR1660*##
QC Sample:	
Volatile Method Blank	VBLK##
Volatile Instrument Blank	VIBLK##
Volatile Storage Blank	VHBLK##
Volatile Leachate Extraction Blank	VLEB##
Semivolatile Method Blank	SBLK##
Semivolatile Leachate Extraction Blank	SLEB##

TABLE 5. CODES FOR LABELING DATA[1,2,3,4,5,6] (CON'T)

Name	Sample Number
Pesticide Method Blank	PBLK##
Pesticide Instrument Blank[1]	PIBLK##
Pesticide Sulfur Blank	PSBLK##
Pesticide Leachate Extraction Blank	PLEB##
Pesticide Laboratory Control Sample[1]	PLCS##
Aroclor Method Blank	ABLK##
Aroclor Instrument Blank[1]	AIBLK##
Aroclor Sulfur Blank	ASBLK##
Aroclor Laboratory Control Sample[1]	ALCS##
Florisil Cleanup Sample[4]	FLO#########
Gel Permeation Chromatograph Cleanup Sample[5]	GPC#########

Footnotes:

[1] When reporting results on forms, "1" or "2" is appended to the EPA Sample Number indicating that the results are from Gas Chromatograph (GC) column (1), [e.g., PLCS01(1) or PLCS01(2) for the second column].

[2] *** = concentration of the standards in µg/L (e.g., 005, 010, etc.). When standard concentrations for semivolatile analysis are in nanograms/microliter (ng/µl) use 005, 010, 020, 040, and 080. Use 0.10, 0.20, 0.40, 0.80, and 1.6 for the SIM analysis of Polynuclear Aromatic Hydrocarbon analytes and pentachlorophenol.

is the identifier with one or two characters or numbers, or a combination of both.

[3] * = standard level for GC/ECD analyses where numbers 1-5 usually represent the standard levels analyzed from low to high as specified in Exhibit D. For example, INDA1## represents the lowest level initial calibration (ICAL) standard and INDA5## for the highest level.

[4] ######### is the Florisil cartridge lot number.

[5] ######### is the GPC column ID.

[6] Instrument QC samples must not be reported as ZZZZZ.

3.3.7.2 These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if A1111 is the lowest (considering both alpha and numeric characters) EPA Sample Number within the SDG, it would be entered in the first EPA Sample Number field. Samples would be listed below it, in ascending sequence – A1111, A1111MS, A1111MSD, AB125, AC111, etc.

3.3.8 "Matrix" is the matrix of the sample. Enter "Soil" for soil/sediment samples and "Water" for aqueous/water and leachate samples, as appropriate.

3.3.9 "Analytical Method" is the method used to analyze the sample. Enter "Trace VOA", "VOA", "SVOA", "SVOA SIM", "PEST", or "ARO", as appropriate.

3.3.10 "Level" is applicable to the soil/sediment samples and blanks analyzed by volatile and semivolatile methods. Enter "LOW" for the low level analysis and "MED" for the medium level analysis.

3.3.11 "Lab Sample ID" is an optional laboratory-generated internal identifier. If the Contractor does not have a Lab Sample ID, this field may be left blank. However, if this identifier is used on any of the forms or accompanying hardcopy data deliverables, it must be reported on all the appropriate forms.

3.3.12 "Sample wt/vol:" is the aliquot amount of the sample used for sample analysis or extraction. Enter the number of grams as measured for soil/sediment samples. Enter the volumes as measured for water samples. Report weights and volumes to three significant figures (e.g., 10.0 g, 955 mL).

3.3.13 "Lab File ID" is the laboratory-generated name of the instrument data system file containing information pertaining to a particular analysis.

3.3.14 "% Solids" is the percent solids of the soil/sediment sample as determined by the procedure in Exhibit D – General Organic Analysis. Report the calculated % Solids to three significant figures.

3.3.15 "Date Extracted" is applicable to samples that have undergone an extraction procedure by the analytical method. The format of MM/DD/YYYY shall be used for the date. When continuous liquid-liquid extraction procedures are used for water samples, enter the date that the procedure was started in the "Date Extracted" field. If separatory funnel, sonication, soxhlet, or pressurized fluid extraction procedures are used, enter the date that the procedure was completed in the "Date Extracted" field.

3.3.16 "Date Analyzed" is common to all samples, blanks, and standards. The format of MM/DD/YYYY shall be used for the date.

3.3.17 "Injection Volume" is volume of the sample extract injected into the GC/MS or GC/ECD instrument for analysis. Report this volume in µL to one decimal place (e.g., 1.0 µL).

3.3.18 "Instrument ID" is the instrument identifier used by the laboratory, particularly on forms containing calibration data. The identifier must include some indication of the manufacturer and/or model of the instrument, and contain additional characters or numbers that differentiate between all instruments of the same type in the laboratory. The instrument identifier must be consistent on all forms within the SDG.

3.3.19 "GC Column" and "ID: (mm)" are two (2) fields used to identify the stationary phase of the GC column and the internal diameter of the GC column in millimeters (mm).

3.3.20 "Extract Volume" is the volume of the final concentrated extract at the completion of the sample extraction process. It is also applicable to medium level sample analysis by the purge-and-trap analytical method where the sample is extracted in methanol. It is entered as the volume measured in the unit of "µL".

3.3.21 "Heated Purge" is applicable to volatiles by purge-and-trap analytical methods. Enter "Y" for heated purge or "N" for ambient temperature purge.

3.3.22 "Extraction Type" is applicable to samples that have undergone extractions per the analytical methods. Enter "SEPF" for separatory funnel, "CLLE" for continuous liquid-liquid extraction without hydrophobic membrane, "CONH" for continuous liquid-liquid extraction with hydrophobic membrane, "SONC" for Sonication Extraction, "SOXH" for Soxhlet Extraction, or "PFEX" for Pressurized Fluid Extraction, as appropriate. For waste dilution, enter "WDIL". For the trace and low/medium volatile analytical methods, enter "PT" for purge-and-trap.

3.3.23 "Cleanup Types" is applicable to samples that have undergone certain cleanup processes by the analytical method. Enter "GPC", "Florisil", "Acid", or "Sulfur" separated by commas, as appropriate.

3.3.24 "Concentration Units" are the units in which the analytical result is reported. Enter "µg/L", "mg/L", or "µg/kg" as appropriate.

3.3.25 "Analyte" is identified in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, and must be reported in the order given in Exhibit C.

3.4 Reporting Forms

3.4.1 SDG Cover Page

3.4.1.1 Purpose

This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager or designee to authorize and release all data and deliverables associated with the SDG.

3.4.1.2 Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.1.2.1 For samples analyzed using this SOW, enter "SOM02.4" for the SOW Number.

3.4.1.2.2 Under column "EPA Sample No.", enter each EPA Sample Number.

3.4.1.2.3 Under column "Lab Sample ID", enter each Laboratory sample identifier.

3.4.1.2.4 Under column "Analysis Method", enter an "X" under each Analytical Method scheduled for analysis for each EPA Sample Number.

3.4.1.2.5 Each SDG Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager’s designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.4.2 Organic Analysis Data Sheet [Form 1A-OR and Form 1B-OR]

3.4.2.1 Purpose

Form 1A-OR is used to tabulate and report sample analysis results for organic target analyte(s) per analytical method (see Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits).

Form 1B-OR is used to report sample analysis results for non-target analytes (e.g., analytes not listed in Exhibit C).

3.4.2.2 Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.2.1 "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the TR/COC Record (i.e., the VTSR).

3.4.2.2.2 "Extract Concentrated" is applicable to samples that have undergone sample cleanup procedures. Enter "Y" for sample extracts concentrated after cleanup; otherwise enter "N".

3.4.2.2.3 "Soil Aliquot (VOA)" is applicable to medium level sample analysis by purge-and-trap analytical method where sample is extracted in methanol. Enter the methanol extract volume added to the reagent water in the purge tube for analysis in the unit of "µL".

3.4.2.2.4 "Purge Volume" is applicable to volatiles. Enter the volume purged in the unit of "mL".

3.4.2.2.5 "pH" is required for aqueous/water samples. Enter the pH determined. Report the pH value for soil/sediment samples, if the measurement is requested.

3.4.2.2.6 "Dilution Factor" is indicative of sample whether it is analyzed undiluted or at dilution. The dilution factor (DF) value shall be reported to one decimal place. Enter 1.0 for an undiluted sample with a dilution factor of 1.

3.4.2.2.7 "Cleanup Factor" is applicable to the sequential cleanup types reported in "Cleanup Types" field. Cleanup factor for each applicable cleanup procedure is determined per Exhibit D and reported in the order of the corresponding cleanup type separated as appropriate by a comma.

3.4.2.2.8 Under column "CAS No.", enter the CAS Number for each analyte as listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits.

3.4.2.2.9 Under column "Concentration", enter for each analyte, the value of the result if the concentration or mass is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions. If the concentration is less than the MDL, enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions. The concentration or mass result shall be reported to two significant figures.

NOTE: For analytes in a sample that require more than one dilution, the compliant result from the least diluted analysis shall be considered as the best analytical result for the sample. For analytes in a sample that require dilution, reanalysis, or re-extraction, when none of the results from these analyses are compliant, the result from the initial analysis shall be considered as the best analytical result for the sample. For non-detected analytes that do not require any further dilution, reanalysis, or re-extraction, the CRQLs from the initial analysis shall be considered as the best analytical result.

3.4.2.2.10 Under column "Q", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included in the SDG Narrative.

3.4.2.2.10.1 The MDL obtained for a given preparation method, analysis method, and instrument shall be used for the qualification of the results for samples associated with that preparation method, analysis method, and instrument.

All values for result, CRQL, and MDL shall be in the same units prior to determining the appropriate qualifier.

3.4.2.2.10.2 Specified entries and their meanings are as follows:

U: The result was less than the MDL.

J: The reported value is less than the CRQL, but greater than or equal to the MDL. This flag is also used for all TICs.

B: The same analyte is found in the associated blank as well.

E: The analyte concentration exceeds the upper limit of the calibration range of the instrument established by the ICAL.

D: The reported value is from a dilution.

C: The identification of the analyte is confirmed by GC/MS when the primary analytical method employed is GC/ECD as appropriate.

A: The reported TIC is a suspected Aldol-condensation product.

N: The reported TIC is has a ≥ 85% match on the mass spectral library search.

P: The reported value is greater than 25% difference between the concentrations determined on two GC columns where applicable.

S: The reported value is determined using a single-point ICAL by GC/ECD analytical method, as appropriate.

H: The reported value is quantitated using peak heights rather than peak areas.

X: The reported value is with laboratory-defined flag. These flags are limited to the letters "X", "Y", and "Z".

3.4.2.2.11 Form 1B-OR shall be submitted for **every trace volatile, low/medium volatile, and semivolatile analysis**, including required dilutions, reanalyses, and blanks, even if no TICs are found. Forms 1B-OR are not required for requested MS/MSD or SIM analyses. See instructions in Exhibit D on TIC identification and quantitation.

3.4.3 Deuterated Monitoring Compound Recovery [Form 2A and 2B-OR] and Surrogate Recovery [Form 2C-OR]

3.4.3.1 Purpose

Form 2A-OR and 2B-OR are used to report the recoveries of the DMCs added to each volatile and semivolatile sample, including dilutions, reanalyses, blanks, and requested MS/MSDs.

Form 2C-OR is used to report the recoveries of the surrogate compounds added to each pesticide and Aroclor sample, blank, LCS, and MS/MSD.

3.4.3.2 Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.3.2.1 For volatile and semivolatile samples, report the %R of each DMC to the nearest whole percentage point on Forms 2A-OR and 2B-OR.

For pesticide and Aroclor samples, report the %R of each surrogate to the nearest whole percentage point on Form 2C-OR.

3.4.3.2.2 Flag each DMC or surrogate recovery outside the QC limits with an asterisk ("*"). The asterisk shall be placed next to the result value.

3.4.3.2.3 Under column "TOT OUT" report the total number of DMC or surrogate recoveries that are outside the QC limits for each sample. If no DMC or surrogate recoveries were outside the limits, enter "0" (zero).

3.4.3.2.4 If the diluted sample is with DMC or surrogate percent recoveries outside the acceptance window, enter the %R values and flag with a "D" where applicable.

3.4.3.2.5 The pesticide and Aroclor surrogate recoveries shall be reported for **both** GC columns. Identify each GC column at the top of Form 2C-OR, entering the stationary phase in the "GC Column" field, and the internal diameter of the column in mm in the "ID" field.

3.4.3.2.6 The assignment of columns as "1" and "2" is left to the discretion of the Contractor when the analyses are performed by simultaneous injection into a two-column GC. The assignment of "GC Column 1" and "GC Column 2" shall be consistent across all reporting forms. If the analysis is **not** performed by simultaneous injection, then the assignment of GC column number shall be based on the chronological order of the two analyses.

3.4.3.2.7 The compound names listed in Exhibit D, Section 17, Table 3 (for Trace Volatiles, Low/Medium Volatiles, and Semivolatiles); Table 10 (for Pesticides); and Table 6 (for Aroclors) for all DMCs or surrogates applicable to the analytical method, shall be reported under each table along with their respective QC limits.

3.4.4 Matrix Spike/Matrix Spike Duplicate Recovery [Form 3A-OR]

3.4.4.1 Purpose

This form is used to report the results of the MS/MSD analyses for all applicable methods.

NOTE: Form 3A-OR shall only be submitted if the analyses of MS/MSD samples are requested or scheduled by the EPA Region. Submit form(s) for each MS/MSD performed.

3.4.4.2 Instructions

Complete the header information according to the instructions in Section 3.3. Include the EPA Sample Number for the Matrix Spike or Matrix Spike Duplicate, without the suffixes "MS" or "MSD". Complete the remainder of the form using the following instructions.

3.4.4.2.1 For pesticides and Aroclors, this form is required for each column. Enter the instrument ID, the stationary phase in the "GC Column" field, and the internal diameter of the column in mm in the "ID" field. The results reported on this order shall be consistent with the information reported on Form 10-OR.

3.4.4.2.2 Under column "SPIKE ADDED", enter the calculated concentration of each spike analyte to the same significant figure as reported for the sample concentration in the appropriate unit.

3.4.4.2.3 Under column "SAMPLE CONCENTRATION", enter the sample concentration of each spike analyte in the original sample in the appropriate unit. If a spike analyte is not detected in the original sample, enter "0" (zero) as the concentration for the analyte.

3.4.4.2.4 Under column "MS CONCENTRATION", enter the concentration of each spike analyte determined in the Matrix Spike sample.

3.4.4.2.5 Under column "MS %R", enter the calculated %R of each spiked analyte in the Matrix Spike sample to the nearest whole percent.

3.4.4.2.6 Under column "QC LIMITS %R", enter the %R limits for each spike analyte as specified in Exhibit D.

3.4.4.2.7 Flag each %R outside the QC limits with an asterisk ("*") next to the %R value in the "MS %R#" column.

3.4.4.2.8 Follow Sections 3.4.4.2.2 through 3.4.4.2.7 to complete the table for the MSD sample.

3.4.4.2.9 Under column "RPD", enter the calculated Relative Percent Difference (RPD) between the Matrix Spike recovery and the Matrix Spike Duplicate recovery. Report the RPD to the nearest whole percent.

3.4.4.2.10 Under column "QC LIMITS", enter the applicable QC limits for %R and RPD respectively as specified in Exhibit D.

3.4.4.2.11 Flag each RPD outside the QC limits with an asterisk ("*") next to the value in the "RPD" column.

3.4.4.2.12 Flag each %R outside the QC limits with an asterisk ("*") next to the value in the "%R" column.

3.4.5 Laboratory Control Sample Recovery [Form 3B-OR]

3.4.5.1 Purpose

This form is used to report the results of the analyses of LCSs for pesticides and Aroclors.

3.4.5.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.5.2.1 "LCS Lot No." is applicable for identifying the LCS solution purchased from a third party. Enter the identification number used by the third party to identify the LCS lot, if available. Leave the field blank if the LCS solution was prepared in-house.

3.4.5.2.2 "Instrument ID", "GC Column", "ID", and "Date Analyzed" fields above each table are entered for each column applicable to pesticides and Aroclors.

3.4.5.2.3 Under column "AMOUNT ADDED", enter the calculated concentration of each spike analyte to the same significant figure as reported for the concentration in the appropriate unit.

3.4.5.2.4 Under column "AMOUNT RECOVERED", enter the concentration of each spike analyte in the LCS sample.

3.4.5.2.5 Under column "%R", enter the calculated %R of each spike analyte to the nearest whole percent.

3.4.5.2.6 Flag each %R value outside the QC limits with an asterisk (*) next to the value.

3.4.5.2.7 Complete the second table according to the instructions above for pesticides and Aroclor secondary column analysis as applicable.

3.4.6 Method Blank Summary [Form 4-OR]

3.4.6.1 Purpose

This form summarizes the samples including dilutions, reanalyses, re-extractions/reanalyses, and the requested MS/MSDs associated with each method blank analysis. The Contractor shall submit the appropriate Form 4-OR for each blank and for all methods. This form is not required for an instrument blank.

3.4.6.2 Instructions

Complete the header information according to the instructions in Section 3.3. The EPA Sample Number entered in the upper right-hand corner shall be the same number entered on Form 1-OR for the blank. Complete the remainder of the form using the following instructions.

3.4.6.2.1 "Instrument ID", "GC Column", "ID", "Date Analyzed", and "Time Analyzed" fields are entered for each column applicable to pesticide and Aroclor analyses. If the analyses were analyzed simultaneously, the information entered here shall be consistent with that on all other applicable forms.

3.4.6.2.2 "Date Analyzed" and "Time Analyzed" fields shall indicate the analysis on both primary and secondary columns (i.e., Time Analyzed: 11:00/11:50, or 11:00, 11:50).

3.4.6.2.3 "Cleanup (Y/N)" is applicable to method blanks that have undergone cleanup procedures. Enter "Y" if any cleanup procedure is performed; otherwise enter "N".

3.4.6.2.4 Under column "EPA SAMPLE No.", enter the EPA Sample Number of samples including LCSs, requested MS/MSDs, storage blanks, and volatile instrument blanks, associated with the reported method blank.

3.4.6.2.5 Under column "LAB SAMPLE ID", enter the Laboratory Sample Identifier for each reported sample under the first column.

3.4.6.2.6 Under column "LAB FILE ID", enter the Laboratory-assigned file Identifier of the analysis for each sample reported under the first column.

3.4.6.2.7 Under column "DATE/TIME ANALYZED", enter the date or time of the analysis of each sample. For volatiles and semivolatiles, enter the date of analysis. For pesticides and Aroclors, enter both analyses times for each column (i.e., 11:00/11:50, or 11:00, 11:50).

3.4.7 GC/MS Instrument Performance Check [Form 5-OR]. This form is not required for Level 2a deliverables.

3.4.7.1 Purpose

This form is used to report the results of the GC/MS instrument performance check (IPC) for the volatile and semivolatile methods, and to summarize the date and time of analyses for samples, including dilutions, reanalyses, calibration standards, blanks, and requested MS/MSDs associated with each analysis of the IPC solution.

3.4.7.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.7.2.1 "BFB/DFTPP" is the compound used to tune the instrument. Enter "BFB" for volatiles or "DFTPP" for semivolatiles.

3.4.7.2.2 "Injection Date" is the date of injection of the IPC solution [4-bromofluorobenzene (BFB) for volatiles or Decafluorotriphenylphosphine (DFTPP) for semivolatiles]. Enter the date as MM/DD/YYYY.

3.4.7.2.3 "Injection Time" is the time of injection of the IPC solution (BFB for volatiles or DFTPP for semivolatiles). Enter time using military time format.

3.4.7.2.4 Under columns "m/e" and "ION ABUNDANCE CRITERIA" in the first table, enter the m/e value and the mass spectral ion abundance criteria for each IPC analysis as specified in Exhibit D.

3.4.7.2.5 Under column "% RELATIVE ABUNDANCE" in the first table, enter the percent relative abundance for the respective ion to the number of significant figures specified in Exhibit D.

NOTE: For both BFB and DFTPP, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak [mass-to-charge ratio (m/z) 95 for BFB and m/z 198 for DFTPP]. Despite this possibility, all ion abundances shall be normalized to the nominal base peaks listed on Form 5-OR.

3.4.7.2.6 All relative abundances shall be reported as a number. If the relative abundance is zero, enter "0", not a dash or other non-numeric character. Where parentheses appear, enter the calculated percentage of the ion abundance of the mass given in Exhibit D.

3.4.7.2.7 Under column "EPA SAMPLE NO." in the second table, enter the EPA Sample Number for the applicable initial calibration, standards, ICVs, opening/closing CCVs, and all samples, including dilutions, reanalyses, blanks, and requested MS/MSDs associated to that IPC in chronological order, by time of analysis (using military time).

3.4.7.2.8 Under columns "LAB SAMPLE ID", "LAB FILE ID", "DATE ANALYZED", and "TIME ANALYZED" in the second table, enter the appropriate information according to Sections 3.4.6.2.4 – 3.4.6.2.7 for the respective analysis reported in the first column.

3.4.8 GC/MS Initial Calibration Data [Form 6A-OR]. This form is not required for Level 2a deliverables.

3.4.8.1 Purpose

This form contains the summary of an initial calibration of the GC/MS analytical methods (volatile and semivolatile). The five-point initial calibration associated to sample analyses is analyzed at the specific concentration levels described in Exhibit D. An initial calibration containing more than five standards may be performed, but only five standards demonstrating the linearity of the calibration at the specified concentration levels in Exhibit D are to be reported on the form.

3.4.8.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.8.2.1 "Calibration Date(s)" is the date(s) of the calibration (entered as MM/DD/YYYY). If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded.

3.4.8.2.2 "Calibration Time(s)" is the time of injections for the first and the last of the analyzed initial calibration standards using military time format.

3.4.8.2.3 "Length" is the GC column length in the unit of "m".

3.4.8.2.4 "Purge Volume" is applicable to volatiles. Enter the volume purged in the unit of "mL".

3.4.8.2.5 "Lab File ID" is the Laboratory File Identifier of the initial calibration standards. Enter the Laboratory File Identifier of the initial calibration standard at the lowest concentration level in the space provided. Enter the Laboratory File Identifier for each initial calibration standard in the order of low to high in the space provided after the "=" sign.

3.4.8.2.6 "RRF" is the Relative Response Factor (RRF) calculated for each target analyte and DMC in the initial calibration standards. Enter the concentration of each of the five standards after "RRF" in the spaces. For example, for a calibration standard concentration at 5.0 µg/L, enter 5.0 after "RRF" in the spaces in the top most row and the appropriate column header.

3.4.8.2.7 Under column "ANALYTE", enter all target analytes and DMC as applicable.

3.4.8.2.7.1 The Trace and Low/Medium Volatile target analytes shall be listed in the same order as in Exhibit D – Trace Concentrations of Volatile Organic Compounds Analysis, Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds, and Exhibit – D – Low/Medium Concentrations of Volatile Organic Compounds Analysis, Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds.

3.4.8.2.7.2 The Semivolatile target analytes shall be listed in the same order as in Exhibit D – Semivolatile Organic Compounds Analysis, Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

3.4.8.2.8 Under columns "$\overline{RRF}$" and "%RSD", enter the calculated mean RRF ($\overline{RRF}$)and the Percent Relative Standard Deviation (%RSD) for each target analyte and DMC reported under the "ANALYTE" column.

3.4.9 Initial Calibration of Single Component Analytes [Form 6B-OR and Form 6C-OR]. These forms are not required for Level 2a deliverables.

3.4.9.1 Purpose

These forms contain the summary of the initial calibration of single component pesticide target analytes and surrogates. For single component pesticide target analtyes and surrogates: mean RTs ($\overline{RT}$s), RT windows, CFs, mean Calibration Factor ($\overline{CF}$), and %RSD are calculated from the five Individual Standard Mixtures A and B or C at the concentrations specified in Exhibit D. Form 6B-OR is for reporting the RTs, $\overline{RT}$s, and RT windows; Form 6C-OR is for reporting $\overline{CF}$s, CFs, and %RSDs.

3.4.9.2 Instructions

Complete Form 6B-OR and Form 6C-OR for **each** GC column used for the five initial individual calibration standards. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.9.2.1 "Level (x CS1)" is the concentration of the five calibration standards as a multiplier of CS1 (Calibration Standard 1). Enter "1.0" for CS1 and 2.0, 4.0, 8.0, and 16.0 for the subsequent CS levels as specified in the Exhibit D in the spaces provided. If the CS5 standard is higher than 16 times CS1, enter the appropriate multiplier to one decimal place.

3.4.9.2.2 "Calibration Date(s)" is the date(s) of the calibration (entered as MM/DD/YYYY). Enter the dates of the first and the last ICAL standard analyses in the entire ICAL sequence [excluding the Resolution Check Standard (RESC), Performance Evaluation Mixture standard (PEM), and instrument blanks]. If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded.

3.4.9.2.3 "Calibration Time(s)" is the time of injections for the first and last of the initial calibration standards using military time. Enter the times of the first and the last ICAL standard analyses in the entire ICAL sequence (excluding the RESC, PEM, and instrument blanks).

3.4.9.2.4 Under column "ANALTYE", enter all applicable target analytes and surrogates in the five initial calibration standards as specified in Exhibit D – Pesticides Analysis, Table 5 – Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates.

3.4.9.2.5 Under column "RT OF STANDARDS" on Form 6B-OR, enter the RT of each applicable target analyte and surrogate determined from each of the initial calibration standards in minutes to the hundredth place.

3.4.9.2.6 Under column "$\overline{RT}$" on Form 6B-OR, enter the calculated $\overline{RT}$ of each target analyte and surrogate determined from each of the five initial calibration standards.

3.4.9.2.7 Under column "RT WINDOW" on Form 6B-OR, enter the calculated RT window for each target analyte and surrogate using the specifications in Exhibit D. The lower limit and upper limit of the RT window shall be entered under "FROM" and "TO", respectively. If there are more than one set of surrogates

present due to Individual Standard Mixture A and B analysis for pesticides, enter only one set of RTs for the surrogates as appropriate.

3.4.9.2.8 Under column "CF OF STANDARDS" on Form 6C-OR, enter the CF of each applicable target analyte and surrogate determined from each of the initial calibration standards.

3.4.9.2.9 Under column "$\overline{CF}$" on Form 6C-OR, enter the calculated $\overline{CF}$ of each target analyte and surrogate determined from each of the five initial calibration standards.

3.4.9.2.10 Under column "%RSD" on Form 6C-OR, enter the calculated %RSD using the specifications in Exhibit D. If there are more than one set of surrogates present due to Individual Standard Mixture A and B analyses for pesticides, enter the appropriate values determined from the same set of surrogates used for the RT windows listed above in Section 3.4.9.2.7.

3.4.10 Initial Calibration of Multicomponent Analytes [Form 6D-OR and 6E-OR]. These forms are not required for Level 2a deliverables.

3.4.10.1 Purpose

These forms contain the summary of the initial calibration of multicomponent pesticide, Toxaphene, and Aroclor target analytes and surrogates. For multicomponent pesticide analyte, Toxaphene, and surrogates: $\overline{RT}$s, RT windows, CFs, $\overline{CF}$s, and %RSDs are calculated from the five Individual Standard Mixtures A and B or C at the concentrations specified in Exhibit D. For the applicable Aroclor target analytes and surrogates, the same parameters are determined from the five initial calibration standards at concentrations specified in Exhibit D. Form 6D-OR is for reporting RTs, $\overline{RT}$s, and RT windows; Form 6E-OR is for reporting CFs, $\overline{CF}$s, and %RSDs.

3.4.10.2 Instructions

Complete Form 6D-OR and Form 6E-OR for each GC column used for the five initial calibration standards of Toxaphene and Aroclors. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.10.2.1 "Level (x CS1)" is the concentration of the five calibration standards as a multiplier of CS1 (Calibration Standard 1). Enter "1.0" for CS1 and 2.0, 4.0, 8.0, and 16.0 for the subsequent CS levels as specified in the Exhibit D in the spaces provided. If the CS5 standard is higher than 16 times CS1, enter the appropriate multiplier to one decimal place.

3.4.10.2.2 "Calibration Date(s)" is the date(s) of the calibration (entered as MM/DD/YYYY). If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded.

3.4.10.2.3 "Calibration Time(s)" is the injection times of the first and last of the initial calibration standards using military time.

3.4.10.2.4 Under column "ANALYTE", enter Toxaphene for the pesticide method and all applicable Aroclor target analytes for the Aroclor method in the five initial calibration standards as specified in Exhibit D – Pesticides Analysis, Table 5 - Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates, or Exhibit D - Aroclors Analysis,

Table 2 - Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors.

3.4.10.2.5 Under column "RT OF STANDARDS" on Form 6D-OR, enter the RT determined for each identified peak from the five individual initial calibration standards for Toxaphene and surrogates of the pesticide method, and each applicable Aroclor target analyte and surrogate of the Aroclor method. RTs shall be entered in minutes to the hundredth place.

3.4.10.2.6 Under column "$\overline{RT}$" on Form 6D-OR, enter the calculated $\overline{RT}$ of each identified peak for Toxaphene and applicable Aroclor target analytes and surrogates determined from each of the five initial calibration standards.

3.4.10.2.7 Under column "RT WINDOW" on Form 6D-OR, enter the calculated RT window for each identified peak of Toxaphene and surrogates as well as applicable Aroclor target analytes and surrogates using the specifications in Exhibit D. The lower limit and upper limit of the RT window shall be entered under "FROM" and "TO", respectively. If Aroclors 1016 and 1260 are analyzed as a combined standard Aroclor 1660, enter the surrogate RT window determined according to the specifications in Exhibit D.

3.4.10.2.8 Under column "CF OF STANDARDS" on Form 6E-OR, enter the CF for each identified peak of Toxaphene and surrogates as well as applicable Aroclor target analytes and surrogates determined from each of the initial calibration standards.

3.4.10.2.9 Under column "$\overline{CF}$" on Form 6E-OR, enter the calculated $\overline{CF}$ for each identified peak of Toxaphene and surrogates as well as applicable Aroclor target analytes and surrogates determined from each of the five initial calibration standards.

3.4.10.2.10 Under column "%RSD" on Form 6E-OR, enter the calculated %RSD for each identified peak using the specifications in Exhibit D. If Aroclors 1016 and 1260 are analyzed as a combined standard Aroclor 1660, enter the appropriate values determined from the same set of surrogates used for the RT windows listed above in Section 3.4.10.2.7.

3.4.11 Initial Calibration (Single Point) of Multicomponent Analytes [Form 6F-OR]. This form is not required for Level 2a deliverables.

3.4.11.1 Purpose

This form contains the summary of single point initial calibration of Toxaphene, applicable Aroclor target analytes, and surrogates. It is for reporting RTs, RT windows, and CFs.

3.4.11.2 Instructions

Complete Form 6F-OR for each GC column used for Toxaphene and Aroclor single point initial calibration standards. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.11.2.1 "Calibration Date(s)" is the date(s) of the calibration (entered as MM/DD/YYYY). If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded.

3.4.11.2.2 "Calibration Time(s)" is the injection times of the first and last of the initial calibration standards using military time.

3.4.11.2.3 Under column "ANALYTE", enter Toxaphene for the pesticide method and all applicable Aroclor target analytes for the Aroclor method in the single point initial calibration standards as specified in Exhibit D. The target analytes shall be listed in the same order as in Exhibit D – Pesticides Analysis, Table 5 – Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates, or Exhibit – D – Aroclors Analysis, Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors.

3.4.11.2.4 Under column "AMOUNT (ng)", enter the amount of the analyte for each standard in the unit of "ng".

3.4.11.2.5 Under column "RT", enter the RT determined for each identified peak for Toxaphene and surrogates of the pesticide method and each applicable Aroclor target analyte and surrogate of the Aroclor method.

3.4.11.2.6 Under column "RT WINDOW", enter the calculated lower and upper limits for each identified peak of Toxaphene and applicable Aroclor target analytes and surrogates determined from the initial calibration standards. The lower and upper limits of the RT window shall be entered under "FROM" and "TO", respectively.

3.4.11.2.7 Under column "CALIBRATION FACTOR", enter the CF for each identified peak of Toxaphene and surrogates as well as applicable Aroclor target analytes and surrogates determined from each of the initial calibration standards.

3.4.12 Resolution Check Summary [Form 6G-OR]. This form is not required for Level 2a deliverables.

3.4.12.1 Purpose

This form contains the summary of the results for RESC, PEM, and Individual Standards A, B, or C (CS3) that shall begin each pesticide initial calibration sequence. This form is also used for reporting the PEM and Individual Standards A, B, or C as CCVs analyzed during the analytical sequence. Form 6G-OR is applicable for each analysis or each GC column used.

3.4.12.2 Instructions

Complete the header information as described in Section 3.3. Use the same assignment of first and second GC columns for reporting the initial calibration standards. Enter the EPA Sample Number for RESC, PEM, or CS3 as specified in Section 3.3.7. If simultaneous injections on a single GC column are used, the EPA Sample Number may be the same for both columns. If simultaneous injections are not used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.

3.4.12.2.1 "Time Analyzed" is the injection time of the applicable RESC, PEM, and CS3 of pesticide initial calibration and the PEM and CS3 continuing calibration standards using military time.

3.4.12.2.2 Under column "ANALYTE", enter each analyte as specified in Exhibit D, in RT order, including both surrogate compounds.

3.4.12.2.3 Under column "RT", enter the RT for each target analyte and surrogate reported under column "ANALYTE".

3.4.12.2.4 Under column "RESOLUTION(%)", enter the calculated percent resolution between each pair of the analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed. Enter the resolutions for the subsequent analyte pairs until all resolutions for the applicable analyte pairs are entered.

3.4.13 Initial Calibration Verification and Continuing Calibration Verification for GC/MS [Form 7A-OR]. This form is not required for Level 2a deliverables.

3.4.13.1 Purpose

This form contains the summary of the ICV and CCV for volatile and semivolatile analyses applicable to GC/MS methods. The Contractor shall submit this form for each associated ICV, and opening and closing CCV relevant to sample analysis.

3.4.13.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.13.2.1 "Time" is the analysis time of the ICV or CCV. Enter time using military time.

3.4.13.2.2 "Init. Calib Date(s)" is for the initial calibration standards associated with the ICV and CCV. Enter dates in the same format as in Section 3.4.8.2.1. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.13.2.3 "EPA Sample No." is for the ICV or CCV. Enter the appropriate EPA Sample Number following the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.13.2.4 "Init. Calib Time(s)" is for the initial calibration standards associated with the ICV and CCV. Enter the corresponding times using military time.

3.4.13.2.5 "Length" is the GC column length in the unit of "m".

3.4.13.2.6 "Purge Volume" is applicable to volatiles. Enter the volume purged in the unit of "mL".

3.4.13.2.7 Under column "ANALYTE", enter the target analytes and DMCs applicable to the specific methods as specified in Exhibit D.

3.4.13.2.7.1 The Trace and Low/Medium Volatile target analytes shall be listed in the same order as in Exhibit D – Trace Concentrations of Volatile Organic Compounds Analysis, Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds, and Exhibit D – Low/Medium Concentrations of Volatile Organic Compounds Analysis, Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds.

3.4.13.2.7.2 The Semivolatile target analytes shall be listed in the same order as in Exhibit D – Semivolatile Organic Compounds Analysis, Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

3.4.13.2.8 Under column "$\overline{RRF}$", enter the $\overline{RRF}$ determined from the associated initial calibration standards for each target analyte and DMC reported under column "ANALYTE".

3.4.13.2.9 The space in "RRF__" is for reporting the concentration of the ICV or CCV applicable to the method as specified in Exhibit D. For example, 50 in the space provided indicates that the concentration of the ICV or CCV is at 50 µg/L as specified for the low/medium volatiles method.

3.4.13.2.10 Under column "RRF__", enter the calculated RRF value for each target analyte and DMC reported under column "ANALYTE" for the ICV or CCV.

3.4.13.2.11 Under column "MIN RRF", enter the appropriate values for the ICV for each target analyte and DMC as specified in Exhibit D. Enter the appropriate values for either opening or closing CCV for each target analyte and DMC as specified in Exhibit D. For a CCV serving as both opening and closing CCV, enter the values for opening CCV.

3.4.13.2.12 Under column "%D", enter the calculated Percent Difference (%D) for each target analyte and DMC reported under column "ANALYTE".

3.4.13.2.13 Under column "MAX %D", enter the appropriate values for the ICV each target analyte and DMC as specified in Exhibit D. Enter the appropriate values for either opening or closing CCV for each target analyte and DMC as specified in Exhibit D. For a CCV serving as both opening and closing CCV, enter the values for opening CCV.

3.4.14 Pesticides Performance Evaluation Mixture Calibration Verification Summary [Form 7B-OR]. This form is not required for Level 2a deliverables.

3.4.14.1 Purpose

This form contains the results of pesticide PEMs that bracket each 12-hour analytical sequence. The Contractor shall submit this form for each PEM associated to the analytical sequence of sample analysis for each GC column.

3.4.14.2 Instructions

Complete Form 7B-OR for each PEM reported on Form 8B-OR. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the forms using the following instructions.

3.4.14.2.1 "Init. Calib Date(s)" is for the initial calibration standards associated with the CCV. Enter the same dates in the same format as in Section 3.4.9.2.2. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.14.2.2 "EPA Sample No. (PEM##)" is for the PEM. Enter the appropriate EPA Sample Number following the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.14.2.3 "Instrument Blank EPA Sample No. (PIBLK##)" and "Instrument Blank Lab Sample ID" are for the instrument blank analyzed right before the CCV in the analytical sequence. Enter the EPA Sample Number and the laboratory sample identifier in the respective fields. For reporting the instrument blank, the laboratory shall follow the naming convention as specified in

Section 3.3, Table 5 – Codes for Labeling Data. Leave the fields blank for the PEM starting the initial calibration sequence.

3.4.14.2.4 "Time Analyzed" are for the time of the instrument blank and PEM pairs. Enter time using military time. Leave the fields blank for instrument blank when the PEM is starting the initial calibration sequence.

3.4.14.2.5 Under column "RT", enter the RT determined in PEM for each target analyte and surrogate reported under column "ANALYTE" in the table.

3.4.14.2.6 Under column "RT window", enter the calculated lower and upper limits for each target analyte and surrogate determined from the associated initial calibration standards. The lower and upper limits of the RT window shall be entered under "FROM" and "TO", respectively.

3.4.14.2.7 Under column "CALC AMOUNT (ng)", enter the calculated amount or each target analyte and surrogate under column "ANALYTE". The values shall be reported to three decimal places with the unit of "ng".

3.4.14.2.8 Under column "NOM AMOUNT", enter the nominal amount for each analyte and surrogate that are under column "ANALYTE". The values shall be reported to three decimal places with the unit of "ng".

3.4.14.2.9 Under column "%D", enter the calculated %D between the calculated amount and nominal amount for each analyte according to Exhibit D.

3.4.14.2.10 "4,4'-DDT %Breakdown", "Endrin %Breakdown", and "Combined %Breakdown" are for the calculated Percent Breakdown (%Breakdown) as specified in Exhibit D.

3.4.15 Continuing Calibration Verification Summary [Form 7C-OR]. This form is not required for Level 2a deliverables.

3.4.15.1 Purpose

This form contains the summary of the applicable CCV for pesticide and Aroclor analyses by GC/ECD methods. The Contractor shall submit this form for each associated opening and closing CCV relevant to sample analysis for each GC column.

3.4.15.2 Instructions

Complete Form 7C-OR for each CCV standard reported on Form 8B-OR. Complete the header information according to the instructions in Section 3.3. Complete the reminder of the forms using the following instructions.

3.4.15.2.1 "Init. Calib Date(s)" is for the initial calibration standards associated to the CCV. Enter the same dates in the same format as in Section 3.4.9.2.2. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.15.2.2 "EPA Sample No." is for the CCV. Enter the appropriate EPA Sample Number following the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.15.2.3 "Instrument Blank EPA Sample No." and "Instrument Blank Lab Sample ID" are for the instrument blank analyzed right before the CCV in the analytical sequence. Enter the EPA Sample Number and the laboratory identifier in the respective fields.

For reporting the instrument blank, the laboratory shall follow the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.15.2.4 "Time Analyzed" are for the time of the instrument blank and CCV (CS3) pairs. Enter time using military time.

3.4.15.2.5 Under column "ANALYTE", enter the target analytes and surrogates applicable to the specific methods as specified in Exhibit D. The target analytes shall be listed in the same order as in Exhibit D – Pesticides Analysis, Table 5 – Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates, or Exhibit D – Aroclors Analysis, Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors.

3.4.15.2.6 Under column "RT", enter the RT determined in the CCV for each target analyte and surrogate reported under column "ANALYTE".

3.4.15.2.7 Under column "RT window", enter the calculated lower and upper limits for each target analyte and surrogate determined from the associated initial calibration standards. The lower and upper limits of the RT window shall be entered under "FROM" and "TO", respectively.

3.4.15.2.8 Under column "$\overline{CF}$", enter the $\overline{CF}$ determined from the associated initial calibration standards for each target analyte and surrogate reported under column "ANALYTE".

3.4.15.2.9 Under column "CF", enter the calculated CF value for each target analyte and surrogate reported under column "ANALYTE" for the CCV.

3.4.15.2.10 Under column "%D", enter the calculated %D value between the CF and $\overline{CF}$ for each target analyte and surrogate reported under column "ANALYTE" for the CCV.

3.4.16 Multi-component Continuing Calibration Verification Summary [Form 7D-OR]. This form is not required for Level 2a deliverables.

3.4.16.1 Purpose

This form contains the summary of the results for Toxaphene and Aroclor CCVs (CS3). The contractor shall submit this form for each associated opening and closing Toxaphene and Aroclor CCVs relevant to sample analysis for each GC column.

3.4.16.2 Instructions

Complete this form for each Toxaphene and Aroclor CCV reported on Form 8B-OR. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the forms using the following instructions.

3.4.16.2.1 "Init. Calib Date(s)" is for the initial calibration standards associated to the CCV. Enter the same dates in the same format as in Section 3.4.9.2.2. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.16.2.2 "Instrument Blank EPA Sample No." and "Instrument Blank Lab Sample ID" are for the instrument blank analyzed right before the Toxaphene or Aroclor CCV in the analytical sequence. Enter the EPA Sample Number and the laboratory identifier in the respective fields. For reporting the instrument blank, the laboratory shall follow the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.16.2.3 "EPA Sample No." is for the Toxaphene and Aroclor CCV. Enter the appropriate EPA Sample Number following the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.16.2.4 "Time Analyzed" are for the time of the instrument blank and CCV (CS3) pairs. Enter time using military time.

3.4.16.2.5 Under column "ANALYTE", enter the target analytes and surrogates applicable to the specific methods as specified in Exhibit D. The target analytes shall be listed in the same order as in Exhibit D – Pesticides Analysis, Table 5 – Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates, or Exhibit D – Aroclors Analysis, Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors.

3.4.16.2.6 Under column "RT", enter the RT determined in the CCV (CS3) for each identified peak of each target analyte and surrogate reported under column "ANALYTE".

3.4.16.2.7 Under column "RT Window", enter the calculated lower and upper limits for each identified peak of each target analyte and surrogate determined from the associated initial calibration standards. The lower and upper limits of the RT window shall be entered for each corresponding peak under "FROM" and "TO", respectively.

3.4.16.2.8 Under column "$\overline{CF}$", enter the $\overline{CF}$ determined from the associated initial calibration standards for each identified peak of each target analyte and surrogate reported under column "ANALYTE".

3.4.16.2.9 Under column "CF", enter the calculated CF value for each identified peak of each target analyte and surrogate reported under column "ANALYTE" for the CCV.

3.4.16.2.10 Under column "%D", enter the calculated %D value between CF and $\overline{CF}$ for each identified peak of each target analyte and surrogate reported under column "ANALYTE" for the CCV.

3.4.17 Internal Standard Area and Retention Time Summary [Form 8A-OR]. This form is not required for Level 2a deliverables.

3.4.17.1 Purpose

This form contains the summary of peak areas and RTs of the internal standards in all volatile and semivolatile calibration standards and samples, including dilutions, reanalyses, and blanks. This form shall be completed for each analytical sequence with either the initial calibration or opening CCV associated to the sample analyses.

3.4.17.2 Instructions

Complete the header information according to Section 3.3. Complete the remainder of the form using the following instructions. If samples are analyzed immediately following an initial calibration, this form shall be completed with the CCV or mid-level calibration standard of the initial calibration (CS3) as the equivalent of an opening CCV. This form can be modified to accommodate more than three internal standards when necessary.

3.4.17.2.1 "Init. Calib Date(s)" is for the initial calibration standards associated to the CCV. Enter dates in the same format as in Section 3.4.8.2.1. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.17.2.2 "EPA Sample No." is for the CCV or the mid-level initial calibration standard CS3. Enter the appropriate EPA Sample Number following the naming convention as specified in Section 3.3, Table 5 – Codes for Labeling Data.

3.4.17.2.3 "Time Analyzed" is for reporting the injection time of the CCV or the mid-level initial calibration standard CS3. Enter time using military time.

3.4.17.2.4 For the "IS AREA" in the header column of the table, report "IS1 AREA" for the first internal standard, "IS2 AREA" for the second internal standard, and "IS3 AREA" for the third internal standard. Additional Form(s) 8A-OR shall be used for additional internal standards, and the number after "IS" incremented accordingly.

3.4.17.2.5 "EPA SAMPLE NO." under the first column is for reporting EPA Sample Numbers for all samples including dilutions, reanalyses, blanks, and requested MS/MSDs that are associated to the CCV or the mid-level initial calibration standard CS3.

3.4.17.2.6 Under column "IS AREA", enter the area responses of the internal standards measured in the CCV or the mid-level initial calibration standard CS3 in the row "12 HOUR STD" in the first column. Enter the calculated upper and lower limits of the areas for each internal standard per specifications in Exhibit D in the rows "UPPER LIMIT" and "LOWER LIMIT", respectively. Enter the area responses of the internal standards measured in each sample reported in the first column.

3.4.17.2.7 Under column "RT", enter the RTs of the internal standards determined in the CCV or the mid-level initial calibration standard CS3 in the row "12 HOUR STD" in the first column. Enter the calculated upper and lower limits of the RTs for each internal standard per specifications in Exhibit D in the rows "UPPER LIMIT" and "LOWER LIMIT", respectively. Enter the RTs of the internal standards measured in each sample reported in the first column.

3.4.17.2.8 If any internal standard area or RT is outside the upper or lower limits as specified in Exhibit D, flag the outlier with an asterisk ("*") to the right of the reported value.

3.4.17.2.9 Report under the table, the compound names listed in Exhibit D, Section 17, Table 9, for all internal standards in the header columns. For example, IS1 = Chlorobenzene-d_5. In addition, report the area and RT upper and lower limits specified in Exhibit D.

3.4.18 Analytical Sequence [Form 8B-OR]. This form is not required for Level 2a deliverables.

3.4.18.1 Purpose

This form contains the summary of the analytical sequence for pesticide and Aroclor analyses. This form shall include the calibration standards, samples, blanks, LCSs, and MS/MSDs within a particular analytical sequence. The form is submitted for each column used for the analyses.

3.4.18.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.18.2.1 "Init. Calib Date(s)" is for the initial calibration standards associated to the CCV. Enter the same dates in the same format as in Section 3.4.9.2.2. Give inclusive dates if the initial calibration is performed over more than one date.

3.4.18.2.2 "Init. Calib Time(s)" is for the initial calibration standards associated with the CCV. Enter the same times in the same format as in Section 3.4.9.2.3.

3.4.18.2.3 "SURROGATE ( ):__" is for the surrogates in the GC/ECD methods and $\overline{RT}$s from initial calibration as specified in Exhibit D. Enter surrogate names, TCX for Tetrachloro-m-xylene and DCB for Decachlorobiphenyl, in the parentheses provided after "SURROGATE 1" and "SURROGATE 2" respectively; and enter the $\overline{RT}$ for each surrogate respectively in the space provided on Form 8B-OR.

3.4.18.2.4 Under column "EPA SAMPLE NO.", enter every analysis associated with a particular analytical sequence as specified in Exhibit D. The Contractor shall include all samples analyzed within the reported analytical sequence. Enter ZZZZZ as the EPA Sample Number to indicate any sample or laboratory QC that is not part of the SDG.

3.4.18.2.5 Under column "LAB FILE ID", enter the unique lab file identifier for each analysis reported in the column "EPA SAMPLE NO.".

3.4.18.2.6 Under column "DATE ANALYZED", enter the date using the format of MM/DD/YYYY.

3.4.18.2.7 Under column "TIME ANALYZED", enter the time of each analysis reported in the column "EPA SAMPLE NO.". Enter time using military time.

3.4.18.2.8 Under columns "SUR 1 RT#" and "SUR 2 RT#", enter the RTs for both surrogates determined in each analysis. Flag any RT value which does not meet the contract requirements by placing an asterisk ("*") to the right of the reported value.

3.4.18.2.9 If the RT cannot be calculated due to interfering peaks, leave the "RT" column blank for that surrogate, enter an asterisk in the last column, and document the problem in the SDG Narrative.

3.4.18.2.10 Multiple forms shall be submitted with consistent header information in order to include all analyses for a particular analytical sequence.

3.4.19 Florisil Cartridge Check [Form 9A-OR]. This form is not required for Level 2a deliverables.

3.4.19.1 Purpose

This form contains the summary of the results for the Florisil Cartridge check analysis with the specific lot of the Florisil Cartridge used for Florisil cleanup.

3.4.19.2 Instructions

Complete the header information according to the instructions in Section 3.3. Enter the Case Number and SDG Number for the current data package, regardless of the original Case for which the cartridge check was performed. Complete the remainder of the form using the following instructions.

3.4.19.2.1 "Florisil Cartridge Lot Number" is for the Lot Number of the Florisil cartridge used for all sample extracts during the Florisil cleanup process.

3.4.19.2.2 Under column "ANALYTE" in the first table, enter the analyte names for the target analytes included in the Florisil Cartridge check solution as specified in Exhibit D.

3.4.19.2.3 Under columns "SPIKE ADDED (ng)" and "SPIKE RECOVERED (ng)" in the upper table, enter the amount of each spike analyte added and the calculated amount of the same analyte recovered with a unit of "ng" as specified in Exhibit D.

3.4.19.2.4 Under column "%R#" in the first table, enter the calculated %R for each spike analyte as specified in Exhibit D. Flag any recovery value that is outside the QC limits as specified in Exhibit D by placing an asterisk ("*") to the right of the reported value.

3.4.19.2.5 Under column "QC LIMITS" in the first table, enter the low and high limits as specified in Exhibit D.

3.4.19.2.6 Under column "EPA SAMPLE NO." in the second table, enter the EPA Sample Number for each sample and blank within the SDG that has undergone the Florisil cleanup procedure using this Florisil Cartridge lot.

3.4.19.2.7 Under column "LAB SAMPLE ID" in the second table, enter the unique laboratory sample identifier for each reported sample in the column under "EPA SAMPLE NO.".

3.4.19.2.8 Under columns "DATE ANALYZED 1" and "DATE ANALYZED 2", enter the dates in the format of DD/MM/YYYY for each reported sample analyzed on two GC columns respectively. Leave "DATE ANALYZED 2" blank if the second column analysis was not performed.

3.4.20 GPC Calibration Verification [Form 9B-OR]. This form is not required for Level 2a deliverables.

3.4.20.1 Purpose

This form contains the summary of the results for GPC Calibration Verification analysis when samples have undergone GPC cleanup.

3.4.20.2 Instructions

Complete the header information according to the instructions in Section 3.3. Enter the Case Number and SDG Number for the current data package, regardless of the original Case for which the cartridge check was performed. Complete the remainder of the form using the following instructions.

3.4.20.2.1 "GPC Column" is for reporting the identifier of the GPC column.

3.4.20.2.2 Under column "ANALYTE" in the first table, enter the analyte names for the target analytes included in the GPC Calibration Verification solution as specified in Exhibit D.

3.4.20.2.3 Under columns "SPIKE ADDED (ng)" and "SPIKE RECOVERED (ng)" in the first table, enter the amount of each spike analyte added and the calculated amount of the same analyte recovered with a unit of "ng" as specified in Exhibit D.

3.4.20.2.4 Under column "%R#" in the first table, enter the calculated %R for each spike analyte as specified in Exhibit D. Flag any recovery value that is outside the QC limits as specified in Exhibit D by placing an asterisk ("*") to the right of the reported value.

3.4.20.2.5 Under column "QC LIMITS" in the first table, enter the low and high limits as specified in Exhibit D.

3.4.20.2.6 Under column "EPA SAMPLE NO." in the second table, enter the EPA Sample Number for each sample and blank within the SDG that has undergone the GPC cleanup process.

3.4.20.2.7 Under column "LAB SAMPLE ID" in the second table, enter the unique laboratory sample identifier for each reported sample in the column under "EPA SAMPLE NO.".

3.4.20.2.8 Under column "GPC CLEANUP DATE", enter the date in the format of MM/DD/YYYY that the sample was subjected to GPC cleanup.

3.4.21 Identification Summary for Single component Analytes [Form 10A-OR]. This form is not required for Level 2a deliverables.

3.4.21.1 Purpose

This form contains the summary of the concentrations of all single component target analytes that are detected on both GC columns. This form shall be submitted for each applicable sample, including dilutions, reanalyses, blanks, LCSs, and MS/MSDs.

3.4.21.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.21.2.1 Under column "ANALYTE", enter the analyte name as appears on Form 1A-OR for each single component pesticide analyte that is positively identified on both columns.

3.4.21.2.2 Under column "RT", enter the RTs of the analytes for each column designated as 1 and 2, respectively. Under column "RT WINDOW", enter the determined lower and upper RT windows for the same analyte from the associated initial calibration standards. The lower and upper limits shall be entered under "FROM" and "TO" for each column designated 1 and 2, respectively.

3.4.21.2.3 Under column "CONCENTRATION", enter the calculated concentration for each column designated 1 or 2, respectively. The concentrations shall be in the same units as that reported on Form 1A-OR.

3.4.21.2.4 Under column "%D", enter the calculated %D between the two concentrations for the designated columns 1 and 2 entered on this form. %D values shall be reported to the same significant figures as specified in Exhibit D. Flag any %D value that is greater than 25% by placing an asterisk ("*") to the right of the reported value.

3.4.21.2.5 Multiple forms shall be submitted with consistent header information in order to include all target analytes that are positively identified on both columns.

3.4.22 Identification Summary for Multicomponent Analytes [Form 10B-OR]. This form is not required for Level 2a deliverables.

3.4.22.1 Purpose

This form contains the summary of the concentrations of the multicomponent target analyte Toxaphene for pesticide analysis and Aroclor target analytes for Aroclor analysis where the reported target analytes are detected on both GC columns. This form shall be submitted for each applicable sample, including dilutions, reanalyses, blanks, LCSs, and MS/MSDs.

3.4.22.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.22.2.1 Under column "ANALYTE", enter the analyte name as appears on Form 1A-OR for each multicomponent pesticide target analyte, Toxaphene, and Aroclor target analytes that are positively identified on both columns.

3.4.22.2.2 Under column "RT", enter the RT of each identified peak for the analytes for each column designated as 1 and 2, respectively.

3.4.22.2.3 Under column "RT WINDOW", enter the determined lower and upper RT windows of each corresponding peak for the same analyte from the associated initial calibration standards. The lower and upper limits shall be entered under "FROM" and "TO" for each column designated 1 and 2, respectively.

3.4.22.2.4 Under column "CONCENTRATION" and sub-column "PEAK", enter the calculated concentration of each identified peak for each column designated 1 or 2, respectively. The concentration values shall be unrounded that will fit the field and in the same units as that reported on Form 1A-OR.

3.4.22.2.5 Under column "CONCENTRATION" and sub-column "MEAN", enter the calculated mean concentration from the peak concentrations for each reported analyte for each column designated 1 and 2, respectively. The mean concentration values shall be rounded to the same significant figures as the values reported on Form 1A-OR.

3.4.22.2.6 Under column "%D", enter the calculated %D between the two concentrations for the designated columns 1 and 2 entered on this form. %D values shall be reported to the same significant figures as specified in Exhibit D. Flag any %D value that is greater than 25% by placing an asterisk ("*") to the right of the reported value.

3.4.22.2.7 Multiple forms shall be submitted with consistent header information in order to include all target analytes that are positively identified on both columns.

3.5 Sample Log-In Sheet [Form DC-1]

3.5.1 Purpose

This form is used to document the receipt and inspection of samples and containers. At least one original Form DC-1 is required for each sample shipping container (e.g., cooler). If the samples in a single sample shipping container must be assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG that has the lowest alpha-numeric number and a copy of Form DC-1 shall be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)", and the location of the original should be noted on the copies.

3.5.2 Instructions

3.5.2.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).

3.5.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.

3.5.2.3 Record the custody seal numbers in Item 2.

3.5.2.4 Open the container, remove the enclosed sample documentation, and record the presence/absence of EPA forms (i.e., TR/COC Records, packing lists) and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.

3.5.2.5 Remove the samples from the shipping container(s), examine the samples and the Sample Tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of Sample Tags in Items 6 and 7.

3.5.2.6 Record the presence or absence of a shipping container temperature indicator bottle in Item 8.

3.5.2.7 Record the shipping container temperature in Item 9. If ice is present, that shall be noted in the "Remarks" column.

3.5.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.

3.5.2.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of shipping container receipt at the laboratory in Items 11 and 12.

3.5.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and the TR/COC Record, and write the sample numbers in the "EPA Sample #" column.

3.5.2.11 Record the appropriate Sample Tags and assigned laboratory numbers, if applicable.

3.5.2.12 Any comments should be made in the "Remarks" column.

3.5.2.13 For Items 1, 3, 4, 6, 7, 8, and 10, circle the appropriate response. Responses can be underlined if this form is completed by automated equipment. Unused columns and spaces shall be crossed out, initialed, and dated.

3.5.2.14 If there are problems observed during receipt or an answer marked with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log and in the SDG Narrative. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.6 Full Organics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

3.6.1 Purpose

The CSF Inventory Sheet is used to record both the inventory of CSF documents and the number of documents in the original Sample Data Package which is sent to the EPA Region.

3.6.2 Instructions

3.6.2.1 Organize all EPA-CSF documents as described in Exhibit B, Sections 2.0 and 3.0. Assemble the documents in Exhibit B, Section 2.0 in the order specified on Form DC-2, and stamp each page with the consecutive number. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX-XXXX, XXXX-XXXX"). For example, when filling out the page numbers for the "Sample Data" section on Form DC-2, enter the page number of the first Form 1A-OR of the sample analysis under the "From" column, and the last page of the raw data of the last sample analysis under the "To" column. The subsequent lines under the "Sample Data" section may be left blank. If there are no documents for a specific document type, enter an "NA" in the empty space.

3.6.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 99, 100, 102, and 103. Category 102 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.6.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential numbers and file the inserts in their logical positions within the CSF (e.g., document to be inserted between pages 6 and 7 shall be numbered as 6a, 6b, 6c, etc.). Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the "Other Records" section of Form DC-2 (e.g., documents to be inserted between pages 6 and 7 shall be numbered as 6a, 6b, 6c, etc.).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

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EXHIBIT B

ORGANIC FORMS

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SDG COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

SOW No.: _____

EPA Sample No.	Lab Sample ID	Analysis Method					
		Trace VOA	Low Med VOA	SVOA	SVOA SIM	PEST	ARO

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 in the SDG Narrative. Release of the data contained in this hardcopy Complete SDG File and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

EPA SAMPLE NO.

FORM 1A-OR
ORGANIC ANALYSIS DATA SHEET
TARGET ANALYTE LIST

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL) _____ Lab File ID: _____

% Solids: _____ Date Received: _____

GC Column: _____ ID: _____(mm) Date Extracted: _____

GC Column: _____ ID: _____(mm) Date Analyzed: _____

Extract Concentrated:(Y/N) _____ Extract Volume: _____(µL)

Soil Aliquot (VOA): _____(µL) Extraction Type: _____

Heated Purge: (Y/N) _____ Injection Volume: _____(µL)

Purge Volume: _____(mL) pH: _____ Dilution Factor: _____

Cleanup Types: _____ Cleanup Factor: _____

Concentration Units (µg/L, mg/L, µg/kg): _____

CAS NO.	ANALYTE	CONCENTRATION	Q

EPA SAMPLE NO.

FORM 1B-OR
ORGANIC ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL) _____ Lab File ID: _____

% Solids: _____ Date Received: _____

GC Column: _____ ID: _____(mm) Date Extracted: _____

Extract Concentrated:(Y/N) _____ Date Analyzed: _____

Soil Aliquot (VOA): _____(µL) Extract Volume: _____(µL)

Heated Purge: (Y/N) _____ Extraction Type: _____

Purge Volume: _____(mL) Injection Volume: _____(µL)

Cleanup Types: _____ pH: _____ Dilution Factor: _____

Concentration Units (µg/L, µg/kg): _____ Cleanup Factor: _____

	CAS No.	ANALYTE	RT	EST. CONC.	Q
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
	E966796[1]	Total Alkanes	N/A		

[1]EPA-designated Registry Number.

FORM 2A-OR
DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____

EPA SAMPLE NO.	DMC1	DMC2	DMC3	DMC4	DMC5	DMC6	DMC7	DMC8	DMC9

FORM 2B-OR
DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____

EPA SAMPLE NO.	DMC10	DMC11	DMC12	DMC13	DMC14	DMC15	DMC16	DMC17	TOT OUT

FORM 2C-OR
SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____

Matrix: _____

GC Column ( ): _____ ID: _____(mm) GC Column ( ): _____ ID: _____(mm)

EPA SAMPLE NO.	SUR 1-1 %R	SUR 1-2 %R	SUR 2-1 %R	SUR 2-2 %R	OTHER (1)	OTHER (2)	TOT OUT

FORM 3A-OR
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____

EPA Sample No. (Matrix Spike/Matrix Spike Duplicate): _____

Instrument ID: _____ GC Column: _____ ID: _____(mm)

Concentration Units (ug/L, mg/L, ug/kg): _____

ANALYTE	SPIKE ADDED	SAMPLE CONCENTRATION	MS CONCENTRATION	MS %R#	QC LIMITS %R

ANALYTE	SPIKE ADDED	MSD CONCENTRATION	MSD %R#	RPD	QC LIMITS	
					RPD	%R

EPA SAMPLE NO.

FORM 3B-OR
LABORATORY CONTROL
SAMPLE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____

Matrix: _____ Lab Sample ID: _____

LCS Lot No.: _____ Date Extracted: _____

Concentration Units (µg/L, mg/L, µg/Kg): _____

Instrument ID ( ): _____ GC Column ( ): _____ ID: _____(mm)

Date Analyzed ( ): _____

ANALYTE	AMOUNT ADDED	AMOUNT RECOVERED	%R	QC LIMITS

Instrument ID ( ): _____ GC Column ( ): _____ ID: _____(mm)

Date Analyzed ( ): _____

ANALYTE	AMOUNT ADDED	AMOUNT RECOVERED	%R	QC LIMITS

EPA SAMPLE NO.

FORM 4-OR
METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Matrix: _____ Lab Sample ID: _____

Instrument ID: _____ Lab File ID: _____

Extraction Type: _____ Date Extracted: _____

GC Column ( ): _____ ID: _____(mm) Date Analyzed: _____

GC Column ( ): _____ ID: _____(mm) Time Analyzed: _____

Heated Purge: (Y/N) __ Cleanup (Y/N): __ Cleanup Types: _____

EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE/TIME ANALYZED

EPA SAMPLE NO.

Form 5-OR
GC/MS Instrument Performance Check

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Lab File ID: _____

Instrument ID: _____ BFB/DFTPP _____

GC Column: _____ ID: _____(mm)

Injection Date: _____ Injection Time: _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )
		( )

EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED

FORM 6A-OR
GC/MS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Instrument ID: _____ Calibration Date(s): _____ _____

GC Column: _____ ID: _____(mm) Calibration Time(s): _____ _____

Length: _____(m)

Heated Purge: (Y/N) _____ Purge Volume: _____(mL)

LAB FILE ID: _____ RRF ____ = _____ RRF ____ = _____

RRF _____ = _____ RRF ____ = _____ RRF ____ = _____

ANALYTE	RRF____	RRF____	RRF____	RRF___	RRF___	$\overline{RRF}$	%RSD

FORM 6B-OR

INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: ______________________________ Contract: ______________________________

Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: _________

Analytical Method: _____________________

Instrument ID: _________________________

Level (x CS1): CS1 ______ CS2 ________ CS3 _________ CS4 ________ CS5 _______

GC Column: __________ ID: ________(mm) Calibration Date(s): ________ _________

Calibration Time(s): ________ _________

ANALYTE	RT OF STANDARDS					$\overline{RT}$	RT WINDOW	
	CS1	CS2	CS3	CS4	CS5		FROM	TO

FORM 6C-OR

INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: ______________________________ Contract: ______________________________

Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: _________

Analytical Method: ____________________

Instrument ID: _________________________

Level (x CS1): CS1 ______ CS2 ________ CS3 _________ CS4 ________ CS5 _______

GC Column: __________ ID: ________(mm) Calibration Date(s): ________ _________

Calibration Time(s): ________ _________

ANALYTE	CF OF STANDARDS					$\overline{CF}$	%RSD
	CS1	CS2	CS3	CS4	CS5		

FORM 6D-OR
INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name: ______________________________ Contract: ______________________________

Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: _________

Analytical Method: ____________________

Instrument ID: _________________________

Level (x CS1): CS1 ______ CS2 ________ CS3 _________ CS4 ________ CS5 _______

GC Column: __________ ID: ________(mm) Calibration Date(s): ________ _________

Calibration Time(s): ________ _________

ANALYTE	PEAK	RT OF STANDARDS					$\overline{RT}$	RT WINDOW	
		CS1	CS2	CS3	CS4	CS5		FROM	TO
	1								
	2								
	3								
	4								
	5								
TCX									
DCB									
	1								
	2								
	3								
	4								
	5								
TCX									
DCB									
	1								
	2								
	3								
	4								
	5								
TCX									
DCB									

FORM 6E-OR
INITIAL CALIBRATION OF MULTICOMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____

Instrument ID: _____

Level (x CS1): CS1 _____ CS2 _____ CS3 _____ CS4 _____ CS5 _____

GC Column: _____ ID: _____(mm) Calibration Date(s): _____ _____

Calibration Time(s): _____ _____

ANALYTE	PEAK	CF OF STANDARDS						
		CS1	CS2	CS3	CS4	CS5	$\overline{CF}$	%RSD
	1							
	2							
	3							
	4							
	5							
TCX								
DCB								
	1							
	2							
	3							
	4							
	5							
TCX								
DCB								
	1							
	2							
	3							
	4							
	5							
TCX								
DCB								

FORM 6F-OR
INITIAL CALIBRATION (SINGLE POINT) OF MULTICOMPONENT ANALYTES

Lab Name: ______________________________ Contract: ______________________________
Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: _________
Analytical Method: ____________________
Instrument ID: _________________________
GC Column: _______ ID: ________(mm) Calibration Date(s): ________ _________
Calibration Time(s): ________ _________

ANALYTE	AMOUNT (ng)	PEAK	RT	RT WINDOW		CALIBRATION FACTOR
				FROM	TO	
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				
		1				
		2				
		3				
		4				
		5				

FORM 6G-OR
RESOLUTION CHECK SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Lab Sample ID ( ): _____

Instrument ID ( ): _____ EPA Sample No.: _____

GC Column ( ): _____ ID: _____(mm) Date Analyzed ( ): _____

Time Analyzed ( ): _____

ANALYTE	RT	RESOLUTION (%)

FORM 7A-OR
INITIAL CALIBRATION VERIFICATION AND CONTINUING CALIBRATION VERIFICATION FOR GC/MS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

Instrument ID: _____ Date Analyzed: _____ Time:_____

Lab File ID: _____ Init. Calib Date(s): _____ _____

EPA Sample No.: _____ Init. Calib Time(s): _____ _____

GC Column: _____ ID: _____(mm) Length: _____(m)

Heated Purge: (Y/N) _____ Purge Volume: _____(mL)

ANALYTE	$\overline{\text{RRF}}$	RRF___	MIN RRF	%D	MAX %D

FORM 7B-OR
PESTICIDE PERFORMANCE EVALUATION MIXTURE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Instrument Blank EPA Sample No. (PIBLK##): _____

Instrument Blank Lab Sample ID: _____

EPA Sample No. (PEM##): _____ Init. Calib Date(s):_____ _____

Lab Sample ID (PEM): _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm) Time Analyzed: _____

Date Analyzed: _____

Time Analyzed: _____

ANALYTE	RT	RT WINDOW		CALC AMOUNT (ng)	NOM AMOUNT (ng)	%D
		FROM	TO			
alpha-BHC						
beta-BHC						
gamma-BHC (Lindane)						
Endrin						
4,4'-DDT						
Methoxychlor						
TCX						
DCB						

4,4'-DDT %Breakdown ( ): _____ Endrin %Breakdown ( ): _____

Combined %Breakdown ( ): _____

FORM 7C-OR
CONTINUING CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Init. Calib Date(s): _____ _____

Instrument Blank EPA Sample No.: _____ Date Analyzed: _____

Instrument Blank Lab ID: _____ Time Analyzed: _____

EPA Sample No.: _____ Date Analyzed: _____

Lab Sample ID: _____ Time Analyzed: _____

GC Column: _____ ID: _____(mm)

ANALYTE	RT	RT WINDOW		$\overline{CF}$	CF	%D
		FROM	TO			

FORM 7D-OR

MULTICOMPONENT CONTINUING CALIBRATION VERIFICATION SUMMARY

Lab Name: ______________________________ Contract: ______________________________

Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: _________

Analytical Method: _____________________ Init. Calib Date(s): _______ _________

Instrument Blank EPA Sample No.: _______ Date Analyzed: _________________________

Instrument Blank Lab ID: _______________ Time Analyzed: _________________________

EPA Sample No.: ________________________ Date Analyzed: _________________________

Lab Sample ID: _________________________ Time Analyzed: _________________________

GC Column: __________ ID: _______(mm)

ANALYTE	PEAK	RETENTION	RT WINDOW		$\overline{CF}$	CF	%D
		RT	FROM	TO			
	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
	1						
	2						
	3						
	4						
	5						
TCX							
DCB							

FORM 8A-OR
INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Level: _____

EPA Sample No.: _____ Lab File ID (Standard): _____

Instrument ID: _____ Init. Calib. Date(s): _____ _____

GC Column: _____ ID: _____(mm) Date Analyzed: _____

Heated Purge: (Y/N) _____ Time Analyzed: _____

	IS AREA	RT	IS AREA	RT	IS AREA	RT
12 HOUR STD						
UPPER LIMIT						
LOWER LIMIT						
EPA SAMPLE NO.						

FORM 8B-OR
ANALYTICAL SEQUENCE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Init. Calib. Date(s): _____ _____

Instrument ID: _____ Init. Calib. Time(s): _____ _____

GC Column: _____ ID: _____(mm)

THE ANALYTICAL SEQUENCE OF BLANKS, SAMPLES, STANDARDS, MS/MSDs, and LCSs IS GIVEN BELOW:

SURROGATE MEAN RT FROM INITIAL CALIBRATION SURROGATE 1 ( ): _____ SURROGATE 2 ( ): _____					
EPA SAMPLE NO.	LAB FILE ID	DATE ANALYZED	TIME ANALYZED	SUR 1 RT#	SUR 2 RT#

Column used to flag RT values with an asterisk.

FORM 9A-OR
FLORISIL CARTRIDGE CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____

Florisil Cartridge Lot Number: _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm)

ANALYTE	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	%R#	QC LIMITS

Column to be used to flag recovery with an asterisk.

THIS CARTRIDGE LOT APPLIES TO THE FOLLOWING SAMPLES, BLANKS, LCSs, AND MS/MSDs:

EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2

FORM 9B-OR
GPC CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____

GPC Column: _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm)

ANALYTE	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	%R#	QC LIMITS

Column to be used to flag recovery with an asterisk.

THIS GPC CALIBRATION VERIFICATION APPLIES TO THE FOLLOWING SAMPLES, BLANKS, LCSs, AND MS/MSDs:

EPA SAMPLE NO.	LAB SAMPLE ID	GPC CLEANUP DATE

EPA SAMPLE NO.

FORM 10A-OR
IDENTIFICATION SUMMARY
FOR SINGLE COMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Lab Sample ID: _____

Instrument ID ( ): _____ Date(s) Analyzed: _____ _____

Instrument ID ( ): _____

GC Column ( ): _____ ID: _____(mm) GC Column ( ): _____ ID: _____(mm)

Concentration Units (µg/L, mg/L, µg/kg): _____

ANALYTE	COL	RT	RT WINDOW		CONCENTRATION	%D
			FROM	TO		
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					

EPA SAMPLE NO.

FORM 10B-OR
IDENTIFICATION SUMMARY
FOR MULTICOMPONENT ANALYTES

Lab Name: ______________________________ Contract: ______________________________

Lab Code: _________ Case No.: ________ MA No.: ____________ SDG No.: __________

Analytical Method: _____________________ Lab Sample ID: __________________________

Instrument ID ( ): _____________________ Date(s) Analyzed: __________ __________

Instrument ID ( ): _____________________

GC Column ( ): _______ ID: _______(mm) GC Column ( ): _______ ID: ________(mm)

Concentration Units (µg/L, mg/L, µg/kg): _____________

ANALYTE	PEAK	RT	RT WINDOW		CONCENTRATION		%D
			FROM	TO	PEAK	MEAN	
__________________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						
__________________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						
__________________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

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

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

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

FORM DC-2
FULL ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LAB NAME _____

LAB CODE _____

CONTRACT NO. _____

CASE NO. _____ SDG NO. _____

MA NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.4)

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
1.	SDG Cover Page				
2.	Traffic Report/Chain of Custody Record(s)				
3.	Sample Log-In Sheet (DC-1)				
4.	CSF Inventory Sheet (DC-2)				
5.	SDG Narrative				
Organic Analysis					
Trace Volatiles					
	Quality Control Summary				
6.	Deuterated Monitoring Compound Recovery (Form 2A-OR and Form 2B-OR)				
7.	Matrix Spike/Matrix Spike Duplicate Recovery (Form 3A-OR) (if requested by the EPA Region)				
8.	Method Blank Summary (Form 4-OR)				
9.	GC/MS Instrument Performance Check (Form 5-OR)				
10.	Internal Standard Area and Retention Summary (Form 8A-OR)				
	Sample Data				
11.	TAL Results – Organic Analysis Data Sheet (Form 1A-OR)				
12.	Tentatively Identified Compounds (Form 1B-OR)				
13.	Raw Data for Each Sample:				
	Reconstructed total ion chromatograms (RICs) for each sample				
	Raw Spectra and background-subtracted mass spectra of target analytes identified				
	Quantitation Reports				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs. FROM	PAGE NOs. TO	CHECK LAB	CHECK REGION
	Mass Spectra of all reported TICs with three best library matches				
	Standards Data (All Instruments)				
14.	GC/MS Initial Calibration Data (Form 6A-OR)				
15.	RICs and Quantitation Reports for all Standards				
16.	Initial Calibration Verification and Continuing Calibration Verification for GC/MS (Form 7A-OR)				
17.	RICs and Quantitation Reports for all Standards				
	Quality Control Data				
18.	Performance Check				
19.	Blank Data				
20.	Matrix Spike/Matrix Spike Duplicate Data (Form 3A-OR) (if requested by the EPA Region)				
21.	Original preparation and analysis forms or copies of preparation and analysis logbook pages (including screening records if applicable)				
Low-Medium Volatiles					
	Quality Control Summary				
22.	Deuterated Monitoring Compound Recovery (Form 2A-OR and Form 2B-OR)				
23.	Matrix Spike/Matrix Spike Duplicate Recovery (Form 3A-OR) (if requested by the EPA Region)				
24.	Method Blank Summary (Form 4-OR)				
25.	GC/MS Instrument Performance Check (Form 5-OR)				
26.	Internal Standard Area and Retention Time Summary (Form 8A-OR)				
	Sample Data				
27.	TAL Results – Organic Analysis Data Sheet (Form 1A-OR)				
28.	Tentatively Identified Compounds (Form 1B-OR)				
29.	Raw Data for Each Sample:				
	Reconstructed total ion chromatograms (RICs) for each sample				
	Raw Spectra and background-subtracted mass spectra of target analytes identified				
	Quantitation Reports				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs. FROM	PAGE NOs. TO	CHECK LAB	CHECK REGION
	Mass Spectra of all reported TICs with three best library matches				
	Standards Data (All Instruments)				
30.	GC/MS Initial Calibration Data (Form 6A-OR)				
31.	RICs and Quantitation Reports for all Standards				
32.	Initial Calibration Verification and Continuing Calibration Verification for GC/MS (Form 7A-OR)				
33.	RICs and Quantitation Reports for all Standards				
	Quality Control Data				
34.	Performance Check				
35.	Blank Data				
36.	Matrix Spike/Matrix Spike Duplicate Data (if requested by the EPA Region)				
37.	Original preparation and analysis forms or copies of preparation and analysis logbook pages (including TCLP/SPLP logs, Percent Solid Determinations logs, and screening records if applicable)				
Semivolatiles					
	Quality Control Summary				
38.	Deuterated Monitoring Compound Recovery (Form 2A-OR and Form 2B-OR)				
39.	Matrix Spike/Matrix Spike Duplicate Recovery (Form 3A-OR) (if requested by the EPA Region)				
40.	Method Blank Summary (Form 4-OR)				
41.	GC/MS Instrument Performance Check (Form 5-OR)				
42.	Internal Standard Area and Retention Time Summary (Form 8A-OR)				
	Sample Data				
43.	TAL Results – Organic Analysis Data Sheet (Form 1A-OR)				
44.	Tentatively Identified Compounds (Form 1B-OR)				
45.	Raw Data for Each Sample:				
	Reconstructed total ion chromatograms (RICs) for each sample				
	Raw Spectra and background-subtracted mass spectra of target analytes identified				
	Quantitation Reports				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs. FROM	PAGE NOs. TO	CHECK LAB	CHECK REGION
	Mass Spectra of all reported TICs with three best library matches				
	GPC chromatograms (if GPC is required)				
	Standards Data (All Instruments)				
46.	GC/MS Initial Calibration Data (Form 6A-OR)				
47.	RICs and Quantitation Reports for all Standards				
48.	Initial Calibration Verification and Continuing Calibration Verification for GC/MS (Form 7A-OR)				
49.	RICs and Quantitation Reports for all Standards				
	Quality Control Data				
50.	Performance Check				
51.	Blank Data				
52.	Matrix Spike/Matrix Spike Duplicate Data (if requested by the EPA Region)				
53.	Raw GPC Data				
54.	For SIM analysis (if requested), at the same sequence as listed above, except for that Form 1B-OR and TIC spectra data which are not required for SIM method.				
55.	Original preparation and analysis forms or copies of preparation and analysis logbook pages (including TCLP/SPLP logs, Percent Solid Determinations logs, and screening records if applicable)				
Pesticides					
	Quality Control Summary				
56.	Surrogate Recovery (Form 2C-OR)				
57.	Matrix Spike/Matrix Spike Duplicate Recovery (Form 3A-OR each columns)				
58.	Laboratory Control Sample Recovery (Form 3B-OR for each column)				
59.	Method Blank Summary (Form 4-OR)				
	Sample Data				
60.	TAL Results – Organic Analysis Data Sheet (Form 1A-OR)				
61.	Raw Data for Each Sample:				
	Chromatograms (Primary Column)				
	Chromatograms (Secondary Column)				
	Quantitation Reports				
	Manual Worksheets				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs. FROM	PAGE NOs. TO	CHECK LAB	CHECK REGION
62.	For Pesticides by GC/MS Confirmation:				
	Copies of raw spectra and copies of background-subtracted mass spectra of target analytes (samples & standards)				
	Standards Data				
63.	Initial Calibration of Single Component Analytes (Form 6B-OR and 6C-OR)				
64.	Initial Calibration of Multicomponent Analytes (Form 6D-OR and 6E-OR)				
65.	Analyte Resolution Summary (Form 6G-OR)				
66.	Pesticide Performance Evaluation Mixture Calibration Verification Summary (Form 7B-OR)				
67.	Continuing Calibration Verification Summary (Form 7C-OR)				
68.	Multicomponent Continuing Calibration Verification Summary (Form 7D-OR)				
69.	Analytical Sequence (Form 8B-OR)				
70.	Florisil Cartridge Check (Form 9A-OR)				
71.	GPC Calibration Verification (Form 9B-OR)				
72.	Identification Summary for Single Component Analytes (Form 10A-OR)				
73.	Identification Summary for Multicomponent Analytes (Form 10B-OR)				
74.	Chromatograms and Quantitation Reports:				
	A printout of Retention Times and corresponding peak areas or peak heights				
	Quality Control Data				
75.	Blank Data				
76.	Matrix Spike/Matrix Spike Duplicate Data				
77.	Laboratory Control Sample				
78.	Raw GPC Data				
79.	Raw Florisil Data				
80.	Original preparation and analysis forms or copies of preparation and analysis logbook pages (including TCLP/SPLP logs, Percent Solid Determinations logs, and screening records if applicable)				

Aroclors

	Quality Control Summary				
81.	Surrogate Recovery (Form 2C-OR)				
82.	Matrix Spike/Matrix Spike Duplicate Summary (Form 3A-OR)				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
83.	Laboratory Control Sample Recovery (Form 3B-OR for each column)				
84.	Method Blank Summary (Form 4-OR)				
	Sample Data				
85.	TAL Results – Organic Analysis Data Sheet (Form 1A-OR)				
86.	Raw Data for Each Sample:				
	Chromatograms (Primary Column)				
	Chromatograms (Secondary Column)				
	Quantitation Reports				
	Manual Worksheets				
87.	For Aroclors by GC/MS Confirmation:				
	Copies of raw spectra and copies of background-subtracted mass spectra of target analytes (samples & standards)				
	Standards Data				
88.	Initial Calibration of Multicomponent Analytes (Form 6D-OR, Form 6E-OR, and Form 6F-OR)				
89.	Multicomponent Continuing Calibration Verification Summary (Form 7D-OR)				
90.	Analytical Sequence (Form 8B-OR)				
91.	Identification Summary for Multicomponent Analytes (Form 10B-OR)				
92.	Chromatograms and data system printouts:				
	A printout of Retention Times and corresponding peak areas or peak heights				
	Quality Control Data				
93.	Blank Data				
94.	Matrix Spike/Matrix Spike Duplicate Data				
95.	Laboratory Control Sample (LCS) Data				
96.	Raw GPC Data (if performed)				
97.	Original preparation and analysis forms or copies of preparation and analysis logbook pages (including Percent Solid Determinations logs and screening records if applicable)				
Additional					
98.	EPA Shipping/Receiving Documents				
	Airbill (No. of Shipments_____)				
	Sample Tags				
	Sample Log-In Sheet (Lab)				

FORM DC-2
ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	PAGE NOs. FROM	TO	CHECK LAB	REGION
99. Misc. Shipping/Receiving Records (list all individual records)				
Communication Logs				
100. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
101. PE/PT Instruction Forms				
102. Other Records (describe or list)				
Communication Logs				

103. Comments:

Completed by:
(CLP Lab) _____ (Signature) _____ (Print Name & Title) _____ (Date)

Audited by:
(EPA) _____ (Signature) _____ (Print Name & Title) _____ (Date)

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EXHIBIT C

ORGANIC TARGET ANALYTE LIST AND
CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: The Contract Required Quantitation Limit (CRQL) values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D.

Changes to the CRQL may be requested under the Modified Analysis (MA) clause in the contract.

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Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits

Table of Contents

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1.0 TRACE VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 1. TRACE VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A]

Analyte Name	CAS Number	CRQLs Trace Water (µg/L)
Dichlorodifluoromethane	75-71-8	0.50
Chloromethane	74-87-3	0.50
Vinyl chloride	75-01-4	0.50
Bromomethane	74-83-9	0.50
Chloroethane	75-00-3	0.50
Trichlorofluoromethane	75-69-4	0.50
1,1-Dichloroethene	75-35-4	0.50
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.50
Acetone	67-64-1	5.0
Carbon disulfide	75-15-0	0.50
Methyl acetate	79-20-9	0.50
Methylene chloride	75-09-2	0.50
trans-1,2-Dichloroethene	156-60-5	0.50
Methyl tert-butyl ether	1634-04-4	0.50
1,1-Dichloroethane	75-34-3	0.50
cis-1,2-Dichloroethene	156-59-2	0.50
2-Butanone	78-93-3	5.0
Bromochloromethane	74-97-5	0.50
Chloroform	67-66-3	0.50
1,1,1-Trichloroethane	71-55-6	0.50
Cyclohexane	110-82-7	0.50
Carbon tetrachloride	56-23-5	0.50
Benzene	71-43-2	0.50
1,2-Dichloroethane	107-06-2	0.50
Trichloroethene	79-01-6	0.50
Methylcyclohexane	108-87-2	0.50
1,2-Dichloropropane	78-87-5	0.50
Bromodichloromethane	75-27-4	0.50
cis-1,3-Dichloropropene	10061-01-5	0.50
4-Methyl-2-pentanone	108-10-1	5.0
Toluene	108-88-3	0.50
trans-1,3-Dichloropropene	10061-02-6	0.50
1,1,2-Trichloroethane	79-00-5	0.50
Tetrachloroethene	127-18-4	0.50

TABLE 1. TRACE VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A] (CON'T)

Analyte Name	CAS Number	CRQLs Trace Water (µg/L)
2-Hexanone	591-78-6	5.0
Dibromochloromethane	124-48-1	0.50
1,2-Dibromoethane	106-93-4	0.50
Chlorobenzene	108-90-7	0.50
Ethylbenzene	100-41-4	0.50
o-Xylene	95-47-6	0.50
m,p-Xylene	179601-23-1	0.50
Styrene	100-42-5	0.50
Bromoform	75-25-2	0.50
Isopropylbenzene	98-82-8	0.50
1,1,2,2-Tetrachloroethane	79-34-5	0.50
1,3-Dichlorobenzene	541-73-1	0.50
1,4-Dichlorobenzene	106-46-7	0.50
1,2-Dichlorobenzene	95-50-1	0.50
1,2-Dibromo-3-chloropropane	96-12-8	0.50
1,2,4-Trichlorobenzene	120-82-1	0.50
1,2,3-Trichlorobenzene	87-61-6	0.50

2.0 LOW/MEDIUM VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 2. LOW/MEDIUM VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A]

Analyte Name	CAS Number	CRQLs Low Water[I] (µg/L)	Low Soil[B] (µg/kg)	Medium Soil[B] (µg/kg)
Dichlorodifluoromethane	75-71-8	5.0	5.0	250
Chloromethane	74-87-3	5.0	5.0	250
Vinyl chloride[C]	75-01-4	5.0	5.0	250
Bromomethane	74-83-9	5.0	5.0	250
Chloroethane	75-00-3	5.0	5.0	250
Trichlorofluoromethane	75-69-4	5.0	5.0	250
1,1-Dichloroethene[C]	75-35-4	5.0	5.0	250
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5.0	5.0	250
Acetone	67-64-1	10	10	500
Carbon disulfide	75-15-0	5.0	5.0	250
Methyl acetate	79-20-9	5.0	5.0	250
Methylene chloride	75-09-2	5.0	5.0	250
trans-1,2-Dichloroethene	156-60-5	5.0	5.0	250
Methyl tert-butyl ether	1634-04-4	5.0	5.0	250
1,1-Dichloroethane	75-34-3	5.0	5.0	250

TABLE 2. LOW/MEDIUM VOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A] (CON'T)

Analyte Name	CAS Number	CRQLs		
		Low Water[I] (µg/L)	Low Soil[B] (µg/kg)	Medium Soil[B] (µg/kg)
cis-1,2-Dichloroethene	156-59-2	5.0	5.0	250
2-Butanone[C]	78-93-3	10	10	500
Bromochloromethane	74-97-5	5.0	5.0	250
Chloroform[C]	67-66-3	5.0	5.0	250
1,1,1-Trichloroethane	71-55-6	5.0	5.0	250
Cyclohexane	110-82-7	5.0	5.0	250
Carbon tetrachloride[C]	56-23-5	5.0	5.0	250
Benzene[C]	71-43-2	5.0	5.0	250
1,2-Dichloroethane[C]	107-06-2	5.0	5.0	250
Trichloroethene[C]	79-01-6	5.0	5.0	250
Methylcyclohexane	108-87-2	5.0	5.0	250
1,2-Dichloropropane	78-87-5	5.0	5.0	250
Bromodichloromethane	75-27-4	5.0	5.0	250
cis-1,3-Dichloropropene	10061-01-5	5.0	5.0	250
4-Methyl-2-pentanone	108-10-1	10	10	500
Toluene	108-88-3	5.0	5.0	250
trans-1,3-Dichloropropene	10061-02-6	5.0	5.0	250
1,1,2-Trichloroethane	79-00-5	5.0	5.0	250
Tetrachloroethene[C]	127-18-4	5.0	5.0	250
2-Hexanone	591-78-6	10	10	500
Dibromochloromethane	124-48-1	5.0	5.0	250
1,2-Dibromoethane	106-93-4	5.0	5.0	250
Chlorobenzene[C]	108-90-7	5.0	5.0	250
Ethylbenzene	100-41-4	5.0	5.0	250
o-Xylene	95-47-6	5.0	5.0	250
m,p-Xylene	179601-23-1	5.0	5.0	250
Styrene	100-42-5	5.0	5.0	250
Bromoform	75-25-2	5.0	5.0	250
Isopropylbenzene	98-82-8	5.0	5.0	250
1,1,2,2-Tetrachloroethane	79-34-5	5.0	5.0	250
1,3-Dichlorobenzene	541-73-1	5.0	5.0	250
1,4-Dichlorobenzene[C]	106-46-7	5.0	5.0	250
1,2-Dichlorobenzene	95-50-1	5.0	5.0	250
1,2-Dibromo-3-chloropropane	96-12-8	5.0	5.0	250
1,2,4-Trichlorobenzene	120-82-1	5.0	5.0	250
1,2,3-Trichlorobenzene	87-61-6	5.0	5.0	250

3.0 SEMIVOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 3. SEMIVOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A]

Analyte Name	CAS Number	CRQLs				
		Low Water By SIM[D] (µg/L)	Low Water[I] (µg/L)	Low Soil By SIM[B,D] (µg/kg)	Low Soil[B] (µg/kg)	Med. Soil[B] (µg/kg)
1,4-Dioxane	123-91-1		2.0		67	2000
Benzaldehyde	100-52-7		10		330	10000
Phenol	108-95-2		10		330	10000
Bis(2-chloroethyl) ether	111-44-4		10		330	10000
2-Chlorophenol	95-57-8		5.0		170	5000
2-Methylphenol[C]	95-48-7		10		330	10000
3-Methylphenol[C,K]	108-39-4		5.0			
2,2'-Oxybis(1-chloropropane)[E]	108-60-1		10		330	10000
Acetophenone	98-86-2		10		330	10000
4-Methylphenol[A,C]	106-44-5		10		330	10000
N-Nitroso-di-n propylamine	621-64-7		5.0		170	5000
Hexachloroethane[C]	67-72-1		5.0		170	5000
Nitrobenzene[C]	98-95-3		5.0		170	5000
Isophorone	78-59-1		5.0		170	5000
2-Nitrophenol	88-75-5		5.0		170	5000
2,4-Dimethylphenol	105-67-9		5.0		170	5000
Bis(2-chloroethoxy)methane	111-91-1		5.0		170	5000
2,4-Dichlorophenol	120-83-2		5.0		170	5000
Naphthalene[F]	91-20-3	0.10	5.0	3.3	170	5000
4-Chloroaniline	106-47-8		10		330	10000
Hexachlorobutadiene[C]	87-68-3		5.0		170	5000
Caprolactam	105-60-2		10		330	10000
4-Chloro-3-methylphenol	59-50-7		5.0		170	5000
2-Methylnaphthalene[F]	91-57-6	0.10	5.0	3.3	170	5000
Hexachlorocyclo-pentadiene	77-47-4		10		330	10000
2,4,6-Trichlorophenol[C]	88-06-2		5.0		170	5000
2,4,5-Trichlorophenol[C]	95-95-4		5.0		170	5000
1,1'-Biphenyl	92-52-4		5.0		170	5000
2-Chloronaphthalene	91-58-7		5.0		170	5000
2-Nitroaniline	88-74-4		5.0		170	5000
Dimethylphthalate	131-11-3		5.0		170	5000
2,6-Dinitrotoluene	606-20-2		5.0		170	5000
Acenaphthylene[F]	208-96-8	0.10	5.0	3.3	170	5000
3-Nitroaniline	99-09-2		10		330	10000
Acenaphthene[F]	83-32-9	0.10	5.0	3.3	170	5000
2,4-Dinitrophenol	51-28-5		10		330	10000
4-Nitrophenol	100-02-7		10		330	10000
Dibenzofuran	132-64-9		5.0		170	5000
2,4-Dinitrotoluene[C]	121-14-2		5.0		170	5000
Diethylphthalate	84-66-2		5.0		170	5000

TABLE 3. SEMIVOLATILES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A] (CON'T)

Analyte Name	CAS Number	CRQLs				
		Low Water By SIM[D] (µg/L)	Low Water[I] (µg/L)	Low Soil By SIM[B,D] (µg/kg)	Low Soil[B] (µg/kg)	Med. Soil[B] (µg/kg)
Fluorene[F]	86-73-7	0.10	5.0	3.3	170	5000
4-Chlorophenyl-phenyl ether	7005-72-3		5.0		170	5000
4-Nitroaniline	100-01-6		10		330	10000
4,6-Dinitro-2-methylphenol	534-52-1		10		330	10000
N-Nitrosodiphenylamine	86-30-6		5.0		170	5000
1,2,4,5-Tetrachlorobenzene	95-94-3		5.0		170	5000
4-Bromophenyl-phenylether	101-55-3		5.0		170	5000
Hexachlorobenzene[C]	118-74-1		5.0		170	5000
Atrazine	1912-24-9		10		330	10000
Pentachlorophenol[C,F]	87-86-5	0.20	10	6.7	330	10000
Phenanthrene[C,F]	85-01-8	0.10	5.0	3.3	170	5000
Anthracene[F]	120-12-7	0.10	5.0	3.3	170	5000
Carbazole	86-74-8		10		330	10000
Di-n-butylphthalate	84-74-2		5.0		170	5000
Fluoranthene[F]	206-44-0	0.10	10	3.3	330	10000
Pyrene[F]	129-00-0	0.10	5.0	3.3	170	5000
Butylbenzylphthalate	85-68-7		5.0		170	5000
3,3'-Dichlorobenzidine	91-94-1		10		330	10000
Benzo(a)anthracene[F]	56-55-3	0.10	5.0	3.3	170	5000
Chrysene[F]	218-01-9	0.10	5.0	3.3	170	5000
Bis(2-ethylhexyl)phthalate	117-81-7		5.0		170	5000
Di-n-octylphthalate	117-84-0		10		330	10000
Benzo(b)fluoranthene[F]	205-99-2	0.10	5.0	3.3	170	5000
Benzo(k)fluoranthene[F]	207-08-9	0.10	5.0	3.3	170	5000
Benzo(a)pyrene[F]	50-32-8	0.10	5.0	3.3	170	5000
Indeno(1,2,3-cd)pyrene[F]	193-39-5	0.10	5.0	3.3	170	5000
Dibenzo(a,h)anthracene[F]	53-70-3	0.10	5.0	3.3	170	5000
Benzo(g,h,i)perylene[F]	191-24-2	0.10	5.0	3.3	170	5000
2,3,4,6-Tetrachlorophenol	58-90-2		5.0		170	5000

4.0 PESTICIDES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 4. PESTICIDES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A,G]

Analyte Name	CAS Number	CRQLs	
		Water (µg/L)	Soil[B] (µg/kg)
alpha-BHC	319-84-6	0.050	1.7
beta-BHC	319-85-7	0.050	1.7
delta-BHC	319-86-8	0.050	1.7
gamma-BHC (Lindane)[C]	58-89-9	0.050	1.7
Heptachlor[C]	76-44-8	0.050	1.7
Aldrin	309-00-2	0.050	1.7
Heptachlor epoxide[C,H]	1024-57-3	0.050	1.7
Endosulfan I	959-98-8	0.050	1.7
Dieldrin	60-57-1	0.10	3.3
4,4'-DDE	72-55-9	0.10	3.3
Endrin[C]	72-20-8	0.10	3.3
Endosulfan II	33213-65-9	0.10	3.3
4,4'-DDD	72-54-8	0.10	3.3
Endosulfan sulfate	1031-07-8	0.10	3.3
4,4'-DDT	50-29-3	0.10	3.3
Methoxychlor[C]	72-43-5	0.50	17
Endrin ketone	53494-70-5	0.10	3.3
Endrin aldehyde	7421-93-4	0.10	3.3
cis-Chlordane[C,J]	5103-71-9	0.050	1.7
trans-Chlordane[C,J]	5103-74-2	0.050	1.7
Toxaphene[C]	8001-35-2	5.0	170

5.0 AROCLORS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 5. AROCLORS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[G]

Analyte Name	CAS Number	CRQLs	
		Water (µg/L)	Soil[B] (µg/kg)
Aroclor-1016	12674-11-2	1.0	33
Aroclor-1221	11104-28-2	1.0	33
Aroclor-1232	11141-16-5	1.0	33
Aroclor-1242	53469-21-9	1.0	33
Aroclor-1248	12672-29-6	1.0	33
Aroclor-1254	11097-69-1	1.0	33
Aroclor-1260	11096-82-5	1.0	33
Aroclor-1262	37324-23-5	1.0	33
Aroclor-1268	11100-14-4	1.0	33

Endnotes:

A. Changes to the Organic Target Analyte List (TAL) (e.g., adding an additional analyte) may be requested under the Modified Analysis clause in the contract.

B. The CRQLs for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D. The moisture content of the samples must be used to adjust the CRQL values appropriately.

C. Toxicity Characteristic Leaching Procedure (TCLP) analyte list. The CRQLs for the TCLP analytes are the "Low Water" CRQLs (Low/Medium Volatiles and Semivolatiles) and the "Water" CRQLs (Pesticides) divided by 1000 in units of mg/L.

D. CRQL for analysis of water and soil samples using SIM technique for PAHs and phenols.

E. Previously known as Bis(2-chloroisopropyl) ether.

F. Target Analyte List for PAHs and Pentachlorophenol analyses request.

G. There is no differentiation between the preparation of low and medium soil samples in this method for analysis.

H. Only the exo_epoxy isomer.

I. Use the water CRQLs for Synthetic Precipitation Leaching Procedures (SPLP).

J. Formerly known as alpha-Chlordane and gamma-Chlordane respectively.

K. Semivolatile target analyte 3-methylphenol is included in this table ONLY for inclusion in the list of TCLP and/or SPLP analytes. Compounds 3-Methylphenol and 4-Methylphenol cannot be separated by the extraction techniques or GC columns used in this method. Therefore, both are represented in this SOW by the 4-methylphenol isomer only. Those data users who wish to analyze 3- and 4-methylphenol separately are encouraged to utilize the CLP-MA process to obtain data for these compounds from the derivatization/GC method (8041A or equivalent).

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EXHIBIT D

INTRODUCTION TO ORGANIC ANALYTICAL METHODS

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Exhibit D – Introduction to Organic Analytical Methods

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1.0 INTRODUCTION

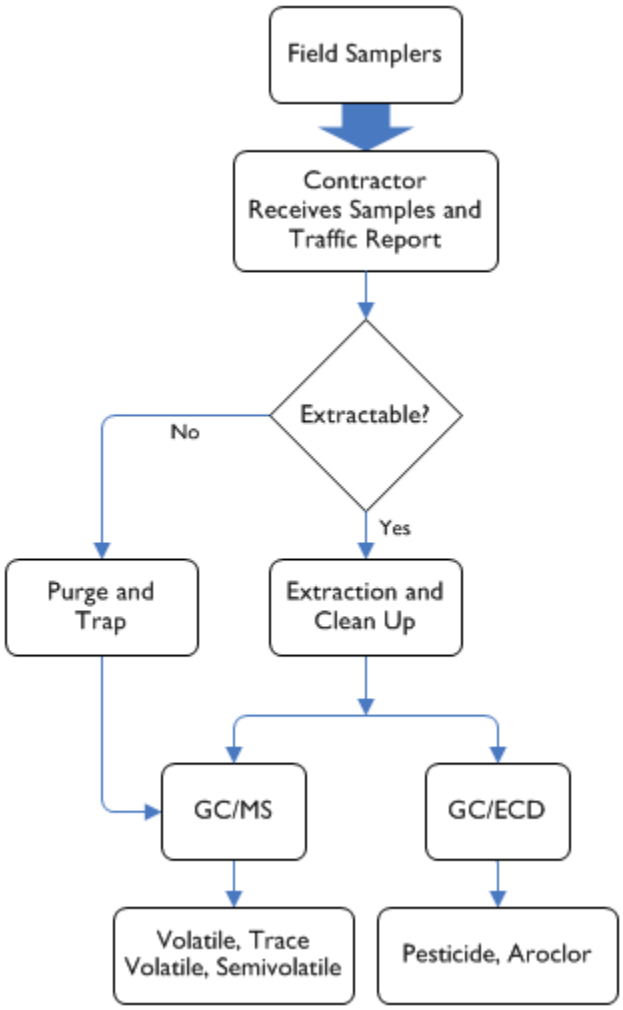
The organic analytical service provides a contractual framework for laboratories. This framework applies the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of trace volatiles, low-medium volatiles, semivolatiles, pesticides, and aroclors in aqueous/water and soil/sediment samples.

The analytical methods that follow are designed to analyze aqueous/water, leachate, and soil/sediment samples from hazardous waste sites for the presence of organic analytes contained in the Organic Target Analyte List (TAL) (see Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits). The organic methods include alternative analysis procedures for some analytes, multiple preparation procedures, and Quality Control (QC) requirements. Analytical techniques in the organic methodologies include Gas Chromatography/Mass Spectrometry (GC/MS) and Gas Chromatography/ Electron Capture Detection (GC/ECD).

2.0 ORGANIC METHODS FLOW CHART

Figure 1 outlines the general analytical scheme the Contractor shall follow in performing standard organic analyses under this contract.

Figure 1 - Organic Methods Flow Chart



3.0 GLASSWARE CLEANING

Laboratory glassware to be used within the organic analyses must be scrupulously cleaned according to the EPA's (SW-846) Chapter Four Organic Analytes, Section 4.1.6, Revision 5, July 2014, or an equivalent procedure. Equivalent procedures are those which meet the Preparation Blank requirements in the Statement of Work (SOW). An electronic version of this manual can found at https://www.epa.gov/sites/production/files/2015-10/documents/chap4_0.pdf.

4.0 STANDARD STOCK SOLUTIONS

Stock solutions to be used for preparing instrument or method standards may be purchased or prepared as described in the individual methods of Exhibit D. Stock solutions that are past the manufacturer's expiration date shall not be used to prepare analytical standards.

5.0 VERIFICATION OF AQUEOUS/WATER SAMPLE CONDITION

At the time of sample receipt, the Contractor shall check the condition of each sample container and its contents and note the condition in a sample receipt log if the condition is not acceptable. The Contractor shall determine if sufficient sample volume has been provided for all tests scheduled and listed on the Traffic Report/Chain of Custody (TR/COC) Record. Containers of water samples for volatile organic analysis should be completely filled without air bubbles. Preservation of samples, if required, should be noted on the label and TR/COC Record. The Contractor shall not adjust the pH of a volatiles sample if preservation is not documented.

6.0 SAMPLE CHARACTERIZATION

6.1 If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact the Sample Management Office (SMO) to apprise them of the type of sample received. SMO will contact the EPA Region.

6.1.1 If all phases of the sample are amenable to analysis, the EPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze one or more of the phases, separately. SMO will provide the EPA Sample Numbers for the additional phases, if required.
- Do not analyze the sample.

6.1.2 If all of the phases are not amenable to analysis (i.e., outside scope), the EPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide the EPA Sample Numbers for the additional phases, if required.
- Do not analyze the sample.

6.1.3 The Contractor shall document the EPA Region's decision in the Sample Delivery Group (SDG) Narrative.

7.0 SAMPLE MIXING

Unless instructed otherwise by the EPA Regional CLP Contracting Officer's Representative (COR), all samples shall be mixed thoroughly prior to aliquoting for extraction. Decant and discard any water layer on a sediment sample. There is no specific procedure provided herein for homogenization of soil/sediment samples; however, an effort shall be made to obtain a representative aliquot. Coarse stones, twigs, or debris that are not representative of the soil/sediment shall be excluded from the aliquot.

8.0 SAMPLE DILUTIONS

The Contractor shall follow the requirements for sample dilutions as described in the individual methods of Exhibit D. The Contractor shall use the least dilution necessary to bring the analyte(s) concentrations within the calibration range. Unless the Contractor can submit proof that dilution was required to obtain valid results, or to avoid damage to Gas Chromatographs or detectors, both diluted and undiluted sample measurements must be contained in the raw data.

8.1.1 The sample and its associated Matrix Spike (MS) and Matrix Spike Duplicate (MSD) shall initially be run at the same dilution.

8.1.2 All volatile water sample dilutions must be made with laboratory reagent water.

8.1.3 All sample extracts must be diluted using the same solvent used in the final sample extract.

9.0 MANUAL INTEGRATIONS

If the Contractor analyzes samples or standards using manual integrations, the Contractor shall clearly identify the manual integrations used to calculate the final sample result and provide the raw data and refer to Exhibit B – Reporting and Deliverables Requirements, Section 2.4 for reporting manual integrations.

10.0 RAW DATA REQUIREMENTS

The Contractor is reminded and cautioned that the collection and reporting of raw data may or may not be referred to within the individual methods of Exhibit D or the Quality Assurance (QA) protocol of Exhibit F - Programmatic Quality Assurance/Quality Control Elements. The raw data deliverable requirements are specified in Exhibit B - Reporting and Deliverables Requirements, Section 2.4. Raw data collected and provided in association with the performance of analyses under this contract shall conform to the appropriate sections of Exhibit B.

11.0 ANALYTICAL STANDARDS REQUIREMENTS

The EPA will not supply analytical reference standards for either direct analytical measurements or the purpose of traceability. All contract laboratories shall be required to prepare, from materials or purchase from private chemical supply companies, those standards necessary to successfully and accurately perform the analyses required in this protocol.

11.1 Preparation of Chemical Standards from the Neat High Purity Bulk Material

11.1.1 If the laboratory cannot obtain analytical reference standards, the laboratory may prepare its own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards. Standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

11.1.2 The chemical standards shall be kept at manufacturer recommended conditions when not being used in the preparation of standard solutions. Proper storage of chemicals is essential to safeguard them from decomposition.

11.1.3 The Contractor is responsible for having analytical documentation demonstrating that the purity of each chemical is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is determined using the following equation:

EQ. 1 Weight of Impure Compound

$$\text{Weight of Impure Chemical} = \frac{\text{weight of pure chemical}}{(\text{percent purity}/100)}$$

WHERE,

Weight of Pure Chemical = That required to prepare a specific volume of a solution standard of a specified concentration.

11.1.4 Logbooks are to be kept for all weighing and dilutions of standards and reagents. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be reviewed and verified by a second person.

11.1.5 All solution standards are to be refrigerated, if required, when not in use.

11.1.6 All solution standards are to be clearly labeled to include the identity of the analyte or analytes, concentration, the standard ID number of the solution, date prepared, solvent, expiration date of the solution, special storage requirements (if any), and initials of the preparer.

11.2 Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria.

11.2.1 Contractors shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

11.2.2 The quality of the reference standards purchased shall be demonstrated statistically and analytically by a method of the supplier's choice.

11.3 Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of the Contractor to maintain the necessary documentation to show that the chemical standards used in the performance of the CLP analysis conform to the requirements previously listed.

11.3.1 In those cases where the documentation is supportive of the analytical results of data packages sent to the Government, such documentation is to be kept on-file by the Contractor for a period of one year.

11.3.2 Upon request by the EPA Regional CLP COR, the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of receipt of the request to the designated recipients.

12.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this SOW has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The Contractor is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of chemicals specified in these methods. A reference file of Material Safety Data Sheets (MSDS) shall be made available to all personnel involved in the chemical analysis.

13.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

14.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with applicable environmental rules and regulations.

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EXHIBIT D

GENERAL ORGANIC ANALYSIS

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Exhibit D –General Organic Analysis

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1.0 SCOPE AND APPLICATION

This Exhibit provides procedures for the use of General Analysis to determine the percent solids of soil/sediment samples, pH, and the leaching of samples by Toxicity Characteristic Leaching Procedure (TCLP) (SW-846 Method 1311) or Synthetic Precipitation Leaching Procedure (SPLP) (SW-846 Method 1312).

2.0 SUMMARY OF METHOD

These methods describe the determination of sample characteristics by gravimetry, electrometry, or the leaching of samples for subsequent analysis by the other analytical methods in this Statement of Work (SOW).

3.0 DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 pH Determination

4.1.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions with a pH <1 may give incorrectly high pH measurements.

4.1.2 Coatings of oily material or particulate matter can impair electrode response. These coatings can generally be removed by gentle wiping or detergent washing followed by rinsing with reagent water. Treatment with 10% HCl may be necessary to remove some films.

4.1.3 Temperature changes can affect measurements. This can be minimized by use of instruments with temperature compensation. The temperature of the sample can change the sample pH. The temperature at which pH measurements are carried out shall be noted.

5.0 SAFETY

See Section 12.0 in Exhibit D - Introduction to Organic Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this SOW is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Percent Solids Determination

6.1.1 Disposable weigh boats with covers

6.1.2 Oven capable of maintaining a temperature of 105°C (±5°C). Oven shall be in a well-ventilated area.

6.1.3 Balance – Top loader, 300 grams (g) capacity with a minimum sensitivity of ±1.0 milligrams (mg)

The balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each balance and be accurate to ±1.0 mg. The masses that are used to check the balances daily must be checked on a monthly basis using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '1' or '2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified every five years or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates that these criteria have been met.

6.2 pH Determinations

6.2.1 pH meter with reference electrode accurate to at least ±0.05 pH units. The pH meter/probe should be equipped with a means of temperature compensation either manually or automatically.

6.2.2 pH paper, wide-range or narrow-range pH paper strip.

6.2.3 Magnetic stirrer with fluoropolymer-coated stir bar.

6.2.4 Beakers – Preferably polyethylene or polytetrafluoroethylene (PTFE).

6.2.5 Various volumetric flasks (Class A) and calibrated pipettes. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.2.6 Thermometer that covers a range of the sample temperature with a minimum accuracy of ±1°C.

6.3 TCLP and SPLP Leaching

6.3.1 Agitation Apparatus – Capable of rotating the extraction vessel(s) in an end-over-end fashion at 30 ±2 rpm.

6.3.2 Extraction Vessels – Jar with sufficient capacity to hold sample and extraction fluid. Vessels shall be constructed of PTFE, stainless steel, or borosilicate glass.

NOTE: PTFE, borosilicate glass, or stainless steel are the only materials suitable when TCLP extracts will be analyzed for organic constituents.

6.3.3 Filters – Borosilicate glass with no binder material with an effective pore size of 0.6–0.8 micrometers (µm). Acid wash with 1N nitric acid prior to use, followed by three consecutive rinses with reagent water [a minimum of 1 Liter (L) per rinse is recommended]. Glass fiber filters are fragile and should be handled with care.

6.3.4 Filtration Device – Capable of exerting pressures up to 50 psi. Use of units having an internal volume of 1.5 L and capable of accommodating a 142 millimeter (mm) filter is recommended.

6.3.5 Beaker – 500 milliliters (mL).

6.3.6 Balance – Any laboratory balance accurate to within ±0.01 grams may be used (all weight measurements are to be within ±0.1 grams). All requirements in Section 6.1.3 shall be met.

6.3.7 Zero-Headspace Extraction (ZHE) Vessel – For volatile analytes, it allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel while effectively excluding headspace. The vessel must be made of inert type 316 stainless steel which will not leach or adsorb sample components. The vessel shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm diameter, 0.6-0.8 µm glass fiber filter. The device contains VITON® O-rings which should be replaced frequently.

6.3.8 An in-line glass fiber filter may be used to filter the material within the ZHE vessel when it is suspected that the glass fiber filter has been ruptured.

NOTE: The ZHE vessel must be free of contaminants and cleaned between TCLP samples. Manufacturer-recommended testing procedures shall be performed to ensure that the apparatus is functioning properly before proceeding with the extraction.

6.3.9 ZHE Extract Collection Devices – TEDLAR® bags or glass, stainless steel, or PTFE gas-tight syringes to collect the initial liquid phase and the final TCLP extract from the ZHE device.

6.3.10 ZHE Extraction Fluid Transfer Devices - Capable of transferring the extraction fluid into the ZHE vessel without changing the nature of the extraction fluid (e.g., a positive displacement or peristaltic pump, a gas-tight syringe).

6.3.11 pH meter with reference electrode accurate to at least ±0.05 units at 25°C. The pH meter/probe should be equipped with a means of temperature compensation either manually or automatically.

6.3.12 Magnetic stirrer with fluoropolymer-coated stir bar.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions. For the preparation of pH buffer solutions, it may be necessary to boil and cool the water prior to use.

7.1.2 Hydrochloric acid, (1N) - Add 83.5 mL conc. hydrochloric acid, 32-38% (specific gravity 1.19) to 400 mL reagent water and dilute to 1 L.

7.1.3 Nitric acid, (1N) – Add 62 mL conc. nitric acid, 67-70% (specific gravity 1.41) to 400 mL reagent water and dilute to 1 L.

7.1.4 Sodium Hydroxide, (1N) - Add 40 g reagent grade NaOH to 400 mL reagent water and dilute to 1 L.

7.1.5 Glacial Acetic Acid – reagent grade.

7.1.6 Sulfuric Acid/Nitric Acid, (60/40 weight percent mixture) – Cautiously mix 60 g (approximately 33 mL) of conc. sulfuric acid, 95-98% (specific gravity 1.84) with 40 g (approximately 28 mL) conc. nitric acid. The Contractor may prepare a more diluted version of this reagent for ease in adjusting extraction fluid pH.

7.1.7 Extraction Fluids

Extraction fluids should be monitored for impurities and the pH checked prior to use. If impurities are found or the pH is not within specifications, the fluid shall be discarded and fresh extraction fluid prepared. Solutions are unbuffered and exact pH may not be attained.

7.1.7.1 TCLP Extraction Fluid #1 – Add 5.7 mL of glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 1N NaOH solution, and dilute to 1 L. The pH of this solution should be 4.93 ±0.05. For ZHE, use TCLP Fluid #1.

7.1.7.2 TCLP Extraction Fluid #2 (do not use Fluid #2 for ZHE) – Dilute 5.7 mL of glacial acetic acid with reagent water to a final volume of 1 L. The pH of this solution should be 2.88 ±0.05.

7.1.7.3 SPLP Extraction Fluid #1 – Use this solution with samples from east of the Mississippi River. Add sufficient 60/40 Sulfuric/Nitric acid solution to reagent water until the pH is 4.20 ±0.05.

7.1.7.4 SPLP Extraction Fluid #2 - Use this solution with samples from west of the Mississippi River. Add sufficient 60/40 Sulfuric/Nitric acid solution to reagent water until the pH is 5.00 ±0.05.

7.1.7.5 SPLP Extraction Fluid #3 – This fluid is reagent water and is used to determine volatiles leachability.

7.1.8 Standard Buffers for pH meter calibration. At a minimum, two standard buffer solutions are required to bracket the expected pH of the samples. The solutions shall be separated by at least three pH units.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. ZHE samples must be collected in PTFE-lined septum-capped vials. All aqueous/water and soil/sediment samples must be iced or refrigerated at a temperature of ≤6°C, but not frozen, from the time of collection until receipt at the laboratory.

8.2 Sample Storage

All aqueous/water and soil/sediment samples must be stored at ≤6°C, but not frozen, from the time of sample receipt until preparation. ZHE samples should be opened just prior to extraction to minimize the loss of volatiles.

8.2.1 Unused Sample Storage

Following preparation for percent solids determination or sample characterization, the remaining unused portion of aqueous/water and soil/sediment samples must be returned to storage at a temperature of ≤6°C, but not frozen, and protected from light. After all applicable leaching procedures and/or extractions have been completed, the remaining unused portion of the aqueous/water and soil/sediment samples must be stored within the laboratory until 60 days after delivery of a complete, reconciled data package to the U.S. Environmental Protection Agency (EPA). The Contractor may store these samples at room temperature. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 Leachate Sample Storage

The remaining unused portion of the preserved TCLP or SPLP leachates must be stored within the laboratory until 180 days after delivery of a complete, reconciled data package to the EPA. The Contractor may store these samples at room temperature.

8.2.3 Container Storage

The Contractor shall retain the empty sample containers for 60 days after delivery of a complete, reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.2.4 Temperature Records

8.2.4.1 The temperature of all sample and sample extract storage refrigerators and freezers shall be recorded daily.

8.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

8.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators and freezers.

8.3 Contract Required Holding Time

The holding time for ZHE extraction of volatile soil samples or waste samples containing $\geq$ 0.5% solids is 10 days from Validated Time of Sample Receipt (VTSR). The holding time for TCLP or SPLP extraction of non-volatile soil samples or waste samples containing $\geq$ 0.5% solids is 10 days from VTSR. The holding time for TCLP or SPLP filtration of aqueous samples is 5 days from VTSR.

9.0 CALIBRATION AND STANDARDIZATION

9.1 pH Meter Calibration

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Each instrument and electrode shall be calibrated at a minimum of two points that bracket the expected pH of the samples. These two points shall be separated by at least three pH units.

Adjust the meter until the readings are within ±0.05 pH units of the buffer solution value.

10.0 PROCEDURE

10.1 Sample Characterization

10.1.1 Percent Solids Determination

Percent Solids determination is based on Standard Method (SM) 2540G, approved 1997.

10.1.1.1 Transfer 5–10 g of sample to a tared weighing boat and record the total weight to the nearest 0.01 g. Sample handling and drying should be conducted in a well-ventilated area.

10.1.1.2 Dry the sample in an oven maintained at 105°C (±5°C) for at least 12 hours, but no more than 24 hours. At the start of drying and at the end of drying, record the oven temperature and date/time.

10.1.1.3 Remove the sample from the oven and allow it to cool in a desiccator.

10.1.1.4 Weigh the sample to the nearest 0.01 g and calculate the percent solids using Equation 1. This value will be used for calculating analytical concentration on a dry weight basis.

EQ. 1 Percent Solids

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

10.1.1.5 For samples scheduled for semivolatile, pesticide, or Aroclor analysis, if the sample contains less than 30% solids, the Contractor shall notify the Sample Management Office (SMO) immediately of the samples impacted. SMO will contact the EPA Region for instructions. This requirement does not apply to 7-day turnaround or Preliminary Results samples. The EPA Region may require the Contractor to do any of the following:

- Use a higher mass of soil/sediment sample (up to 50 g).
- Separate the phases by centrifugation or settling and analyze one or more of the phases separately. SMO will provide EPA Sample Numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2 pH Determinations

10.1.2.1 Aqueous/Water pH Determination

The determination of pH is required for all aqueous/water samples at the time of the receipt at the laboratory or after sample aliquots have been taken. The Contractor shall follow the procedures for pH measurement based on the EPA SW-846 Method 9041A, Revision 1, July 1992 (pH paper) or the EPA SW-846 Method 9040C, Revision 3, November 2004 [electrometric method (i.e., pH meter and electronic hand-held pen)].

10.1.2.1.1 pH Measurement by pH Paper

Place one or two drops of sample on the pH paper and record the pH for the sample.

10.1.2.1.2 pH Measurement by Electrometric Method

10.1.2.1.2.1 Transfer a sufficient volume of sample to a beaker to cover the sensing elements of the electrode(s) and to give adequate clearance for the magnetic stirring bar. The sample shall not be diluted.

10.1.2.1.2.2 If the sample temperature differs by more than 2°C from the temperature of the buffer solutions used to standardize the meter, the measured pH values must be corrected.

10.1.2.1.2.3 After rinsing and gently wiping the electrode(s) if necessary, immerse the electrode(s) in the sample beaker and stir at a constant rate to provide homogeneity and suspension of solids. The rate of stirring should minimize the air transfer rate at the air/water interface. Record the sample pH and the temperature. Repeat measurements on successive volumes of sample until values differ by less than 0.1 pH units.

10.1.2.2 Soil/Sediment pH Determination

The determination of pH for soil/sediment samples is not required as a routine procedure to be completed at the laboratory. However, if requested at the time of scheduling, the Contractor shall follow the procedures based on the EPA SW-846 Method 9045D, Revision 4, November 2004 to determine the pH by electrometric method (i.e., pH meter or electronic hand-held pen).

10.1.2.2.1 Transfer 20 g of well-mixed sample to a 50 mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 1 hour. Additional water may be added if the soils are hygroscopic or contain large amounts of salts.

10.1.2.2.2 Let the soil suspension stand for at least 1 hour to allow most of the suspended clays to settle. Difficult samples may be filtered or centrifuged to separate the aqueous layer for pH determination. If the supernatant is biphasic, decant the oily phase and measure the pH of the aqueous phase.

10.1.2.2.3 Measure and record the pH for the sample.

10.1.2.2.4 Measure and record the temperature for the sample. If the sample temperature differs by more than 2°C from the temperature of the buffer solutions used to standardize the meter, the measured pH values must be corrected.

10.2 TCLP and SPLP Extraction Procedures

Extraction methods are based on EPA SW-846 Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), Revision 0, July 1992 or EPA SW-846 Method 1312, Synthetic Precipitation Leaching Procedure (SPLP), Revision 0, September 1994.

TCLP vessel and devices must be free of contaminants and cleaned between TCLP samples. Testing procedures shall be performed to ensure the apparatus is functioning properly before proceeding with the extraction.

10.2.1 Preliminary Evaluation

Perform preliminary evaluation on a minimum 100 g sample aliquot. This aliquot will not undergo extraction. These preliminary evaluations include: (1) determination of percent solids by pressure filtration; (2) determination of whether the sample contains insignificant (<0.5%) solids and is therefore its own extract after filtration; (3) determination of whether the solid portion of the sample requires particle size reduction; and for TCLP samples, (4) determination of the appropriate extraction fluid.

10.2.1.1 Preliminary determination of percent solids - For these samples, percent solids is defined as that fraction of a sample (as a percentage of the total sample) from which no liquid can be forced out by applied pressure.

10.2.1.1.1 If a sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids), proceed to extraction.

10.2.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required.

10.2.1.1.2.1 Pre-weigh the filter and the container that will receive the filtrate.

10.2.1.1.2.2 Assemble the filter holder and filter per the manufacturer’s instructions. Place the filter on the support screen and secure.

10.2.1.1.2.3 Weigh out at least 100 g of the sample and record the weight.

10.2.1.1.2.4 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered, followed by filtration of the solid portion of the sample through the same filtration system.

10.2.1.1.2.5 Quantitatively transfer the sample to the filter holder (both liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at a temperature of ≤6°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm to room temperature in the device before filtering. If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section

10.2.1.1.2.8 to determine the weight of sample that will be filtered.

10.2.1.1.2.6 Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration. Note that instantaneous application of high pressure can damage the filter and may cause premature plugging.

10.2.1.1.2.7 The material retained on the filter is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Note that certain oily wastes and paint wastes will contain material that appears to be a liquid. However, this material may not filter under pressure filtration. In this case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

10.2.1.1.2.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the sample. Record the weights of the liquid and solid phases. Calculate the percent solids using the following equation:

EQ. 2 Extraction Percent Solids

$$\% \text{ Solids} = \frac{\text{Weight of solid}}{\text{Total Weight of Sample}} \times 100$$

10.2.1.1.2.9 If the percent solids determined in Equation 2 is equal to or greater than 0.5%, then proceed to Section 10.2.1.3 to determine whether the solid material requires particle size reduction.

10.2.1.1.2.10 If it is noticed that a small amount of the filtrate is entrained in wetting of the filter, remove the solid phase and filter from the filtration apparatus. Dry the filter and solid phase at 100°C (±20°C) until two successive weighings yield the same value (within ±1%) and record the weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

10.2.1.1.2.11 Calculate the Percent Dry Solids using the following equation:

EQ. 3 Percent Dry Solids

$$\text{Percent Dry Solids} = \frac{\text{(Wt. of dry waste and filter) - Tared wt. of filter}}{\text{Initial wt. of waste}} \times 100$$

10.2.1.2 If the percent dry solids is less than 0.5%, then treat the filtrate as the extract. Store this extract at a temperature of ≤6°C.

10.2.1.3 To determine if particle size reduction is required, using a fresh portion of sample, examine the solid portion for particle size. If the material is less than 1 centimeter (cm) in its narrowest dimension (i.e., is capable of passing through a 9.5 mm standard sieve), no particle size reduction is required. Otherwise, prepare the solid portion for extraction by crushing, cutting, or grinding the sample to meet the above criterion.

10.2.1.3.1 Special precautions must be taken when processing solid samples for organic volatiles extraction. Wastes and appropriate reduction equipment should be refrigerated, if possible, to ≤ 6°C prior to particle size reduction. The means used to affect particle size reduction must not generate heat. If reduction the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be minimized.

10.2.1.4 For samples that are scheduled for extraction with percent solids greater than 0.5%, the appropriate extraction fluid is determined as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1. Therefore, if TCLP extraction only for volatiles is required, proceed to Section 10.2.3.

10.2.1.4.1 For samples scheduled for TCLP extraction of non-volatile constituents, remove a small aliquot of the sample and reduce the particle size to less than 1 mm. Transfer 5 g of this material to a 500 mL beaker or Erlenmeyer flask.

10.2.1.4.1.1 Add 96.5 mL of reagent water, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH.

10.2.1.4.1.1.1 If the pH is less than 5.0, use TCLP Extraction Fluid #1 (Section 7.1.7.1).

10.2.1.4.1.1.2 If the pH is greater than or equal to 5.0, add 3.5 mL 1N HCl (Section 7.1.2), slurry briefly, cover with the watchglass, and heat to 50°C for 10 minutes. Let the solution cool to room temperature and measure the pH. If the pH is less than 5.0, use TCLP Extraction Fluid #1 (Section 7.1.7.1), otherwise use TCLP Extraction Fluid #2 (Section 7.1.7.2).

NOTE: DO NOT USE FLUID #2 FOR ZHE SAMPLES.

10.2.1.4.2 Use the SPLP extraction fluid appropriate to the information provided on the scheduling document.

10.2.1.4.2.1 For soil samples from east of the Mississippi River, use SPLP Extraction Fluid #1. For samples west of the Mississippi River, use SPLP Extraction Fluid #2.

10.2.1.4.2.2 For samples scheduled for SPLP ZHE extraction, use SPLP Extraction Fluid #3 (Section 7.1.7.5).

10.2.2 TCLP Sample Extraction

Follow this procedure for TCLP leachates that will be analyzed for non-volatile organic target analytes. For volatile organic analysis, use ZHE in Section 10.2.3.

10.2.2.1 A minimum sample size of 100 g is required; however, enough solids shall be extracted to yield a sufficient volume of extract to support all required analyses. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, and whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid. See Section 10.2.2.3 to determine the approximate amount of extract that will be generated for a given mass with the percent solids determined in Section 10.2.1.1.2.11.

10.2.2.1.1 If the sample is 100% solids, then weigh out 100 g of sample and proceed to Section 10.2.2.3.

10.2.2.1.2 If the sample is less than 0.5% solids, filter enough sample to yield a sufficient volume of extract to support all required analyses if the preliminary percent solids determination did not yield sufficient volume.

10.2.2.1.3 For multiphasic samples with percent solids greater than or equal to 0.5%, but less than 100%, weigh out enough sample to generate a sufficient volume of extract to support all required analyses. Filter the sample using the procedure described in Section 10.2.1. Store the filtrate at ≤6°C, but not frozen.

10.2.2.2 Prepare the solid portion of the sample for extraction by reducing the particle size as described in Section 10.2.1.3. Quantitatively transfer the material into an extractor bottle and include the filter used to separate the initial liquid from the solid phase.

10.2.2.3 Determine the amount of extraction fluid to add to the extractor bottle using the following equation:

EQ. 4 Weight of Extraction Fluid

$$\text{Weight of Extraction Fluid} = \frac{20 \times \%\ \text{solids} \times \text{Weight of sample filtered}}{100}$$

10.2.2.4 Add this amount of the appropriate extraction fluid (Section 10.2.1.4) to the extractor bottle. Close the bottle tightly (Teflon tape may be used to ensure a tight seal) and secure it in the rotary agitation apparatus. Rotate the samples at 30 rpm (±2 rpm) for 18 hours (±2 hours). Maintain a temperature of 23°C (±2°C) in room where extraction is performed.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of samples (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

10.2.2.4.1 Following the 18-hour extraction, separate the material in the extractor bottle into its component liquid and solid phase by filtering through a new glass filter as described in Section 10.2.1.1. For the final filtration of the extract, the glass fiber filter may be changed as necessary during filtration.

10.2.2.4.2 If the sample was 100% solids, this filtered liquid is the extract.

10.2.2.4.3 For multiphasic samples, combine this extract with the filtrate generated in Section 10.2.2.1.3 if the two liquids are miscible. If the two liquids are not miscible, they shall be prepared and analyzed separately and the analytical results mathematically combined.

10.2.2.4.4 Record the pH of the final extract. If organic and inorganic analyses are required on the sample, separate approximately 3/4 of the sample extraction fluid for organic analysis and store in an amber glass bottle.

10.2.2.4.5 DO NOT ACIDIFY OR PRESERVE ANY PORTION OF AN EXTRACT INTENDED FOR ORGANIC ANALYSIS. Do not acidify any non-aqueous portion of the sample.

CAUTION: Nitric acid should not be mixed with organic compounds because of the possibility of dangerous reaction.

10.2.3 Zero Headspace Extraction

Use ZHE for the TCLP sample extraction for analysis of volatile organic target analytes. For non-volatile organic target analytes, follow the TCLP Sample Extraction procedure in Section 10.2.2. Follow manufacturer's instructions for operation of the ZHE apparatus.

10.2.3.1 Maintaining the ZHE Apparatus

10.2.3.1.1 The ZHE vessel and devices must be free of contaminants and cleaned between TCLP samples. Manufacturer-recommended testing procedures shall be performed to ensure the apparatus is functioning properly before proceeding with the extraction.

10.2.3.1.2 Disassemble and clean the ZHE parts using laboratory detergent. Rinse with methanol and water until there is no visible contamination when surfaces are wiped with a clean paper towel. Bake ZHE metal parts overnight in an oven at 170°C.

10.2.3.1.3 Reassemble the ZHE and check that it is clean by adding 250 mL of laboratory reagent water, pressurizing the unit, and tumbling for about 1 hour, making sure it is pressure tight. Collect the laboratory reagent water and analyze as a check sample by Gas Chromatography/Mass Spectrometry (GC/MS) to determine if the ZHE is clean. If any target analytes are detected, disassemble the ZHE and repeat the cleaning.

10.2.3.1.4 Record the date, time, and results of each cleaning check in a ZHE laboratory log.

10.2.3.1.5 Disassemble, clean, and check the ZHE, and allow the parts to air dry. Cover the ZHE components in aluminum foil and store in the volatile organics analysis laboratory until use.

10.2.3.1.6 Check the ZHE for leaks after every extraction. Pressurize the ZHE to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the ZHE device does not have a pressure gauge, submerge the pressurized ZHE in water and check for air leaks. If the ZHE is leaking, check all fittings, inspect O-rings, and replace if necessary. Retest the device. If the leakage cannot be solved, the ZHE should be taken off-line and sent to the manufacturer for repairs.

10.2.3.1.7 The piston within the ZHE device must be movable with approximately 15 psi or less. If more than 15 psi is required to move the piston, replace the O-rings. If this does not free up the piston, the ZHE should be taken off-line and sent to the manufacturer for repairs.

10.2.3.2 Zero Headspace Extraction of Volatile Compounds

10.2.3.2.1 The ZHE has a 500 mL internal capacity and accommodates a maximum of 25 g solid based on the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase (fraction of sample from which no additional liquid may be forced out when 50 psi is applied).

10.2.3.2.2 Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

10.2.3.2.3 Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary.

10.2.3.2.4 Pre-weigh the evacuated filtrate collection container and set aside.

10.2.3.2.5 Place the ZHE piston within the body of the ZHE. Adjust the height of the piston to minimize the travel distance once the ZHE is charged with the sample. Secure bottom flanges. Secure the glass fiber filter between the support screens and set top flanges according to manufacturer's instructions.

10.2.3.2.6 If the sample is 100% solids, then weigh a maximum of 25 g and proceed to Section 10.2.3.2.9.

10.2.3.2.7 If the sample is less than 0.5% solids, filter enough sample to yield a sufficient volume of extract to support all volatile analyses required.

For samples containing ≥0.5% solids, use the percent solids determination in Section 10.2.1.1.2.8 to determine the sample size to add to the ZHE using the following equation:

EQ. 5 Sample Size

$$\text{Weight} = \frac{25}{\%\ \text{solids}} \times 100$$

10.2.3.2.8 For multiphasic samples, weigh out enough sample to generate a sufficient volume of extract to support all required analyses. Filter the sample using the procedure described in Sections 10.2.1.1.2.7 – 10.2.1.1.2.9. Store the filtrate at a temperature of ≤6°C.

10.2.3.2.9 Prepare the solid portion of the sample for extraction by reducing the particle size as described in Section 10.2.1.3.

10.2.3.2.10 Determine the amount of TCLP Extraction Fluid #1 to add to the ZHE using the following calculation:

EQ. 6 Weight of Extraction Fluid

$$\text{Weight of Extraction Fluid} = \frac{20 \times \%\ \text{solids} \times \text{Weight of sample filtered}}{100}$$

10.2.3.2.11 Quickly transfer the entire sample (liquid and solid phases) quantitatively to the ZHE. Secure the filter and support screens onto the top flange of the device. Secure the top flange. Tighten all ZHE fittings according to the manufacturer's instructions. Place the ZHE device in vertical position with the gas inlet/outlet flange on the bottom. Do not attach the extract collection device to the top plate at this stage.

10.2.3.2.12 Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

10.2.3.2.13 Attach the evacuated pre-weighed filtrate collection container (Section 10.2.3.2.4) to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

10.2.3.2.14 The material in the ZHE is defined as the solid phase of the sample and the filtrate is defined as the liquid phase.

NOTE: Oily samples and some paint samples may contain material that appears to be a liquid. If after applying pressure filtration the material will not filter, it shall be defined as a solid and is carried through the TCLP extraction as a solid. If the original sample contained <0.5% dry solids, this filtrate shall be defined as the TCLP extract and analyzed directly.

10.2.3.2.15 With the ZHE device in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. Add the appropriate amount of the TCLP Extraction Fluid #1 to solid material within the ZHE device.

10.2.3.2.16 The line used must contain fresh TCLP Extraction Fluid #1 and shall be pre-flushed with fluid to eliminate any air in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue introducing extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

10.2.3.2.17 Close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. The bleeding must be done quickly and stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

10.2.3.2.18 Secure the ZHE device in the rotary agitation apparatus. Rotate the samples at 30 rpm (±2 rpm) for 18 hours (±2 hours). Maintain a temperature of 23°C (±2°C) in room where extraction is performed.

10.2.3.2.19 Following the 18-hour extraction period, check that the ZHE is not leaking by quickly opening and closing the gas inlet/outlet valve, and noting the escape of gas. There will be no escape of gas if the device is leaking. If the ZHE device was leaking, perform the extraction again with a new sample.

10.2.3.2.20 If the pressure within the device has been maintained, the material in the extractor vessel shall be once again separated into its component liquid and solid phases. If the sample contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container holding the initial liquid phase of the sample.

10.2.3.2.21 A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed. All extracts shall be filtered and collected in the collection container if the extract is multiphasic, or if the sample contained an initial liquid phase.

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

If the original sample contained no initial liquid phase, the filtered liquid material obtained from ZHE procedure shall be defined as the TCLP extract. If the sample contained an initial liquid, the filtered liquid material obtained from the ZHE procedure and the initial liquid phase shall be collectively defined as the TCLP extract.

10.2.3.2.22 Following collection of the TCLP extract, immediately prepare the extract for analysis, and store with minimal headspace at a temperature of ≤6°C until analyzed.

If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

EQ. 7 Final Concentration

$$\text{Final Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

WHERE,

V_1 = The volume of the first phases (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

10.2.4 SPLP Sample Extraction

The Contractor shall follow the procedures in Section 10.2.2 using the appropriate extraction fluid specified in Section 10.2.1.4.2.

11.0 DATA ANALYSIS AND CALCULATIONS

See individual procedures in Section 11.0 for data analysis and calculations.

12.0 QUALITY CONTROL

12.1 Leachate Extraction Blank

12.1.1 The Leachate Extraction Blank (LEB) shall contain all the reagents and in the same volumes as used in extracting the samples. The LEB shall be carried through the complete extraction procedure.

12.1.2 At least one LEB, consisting of the appropriate extraction fluid processed through the extraction procedure, shall be extracted with every SDG scheduled for TCLP or SPLP.

12.1.3 Each Complete SDG File (CSF) shall contain the results of all LEB analyses associated with the samples in that SDG.

12.1.4 The LEB(s) result(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination.

12.1.5 Under no circumstances should the LEB be analyzed at a dilution.

12.2 Summary of Quality Control Operations

The Quality Control (QC) operations performed are summarized in Section 17.0, Table 1 – Quality Control Operations.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 in Exhibit D - Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 in Exhibit D - Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1311, Revision 0, Update III, July 1992.

16.2 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1312, Revision 0, Update III, September 1994.

16.3 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 9040C, Revision 3, November 2004.

16.4 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 9041A, Revision 1, July 1992.

16.5 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 9045D, Revision 4, November 2004.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. QUALITY CONTROL OPERATIONS

QC Operation	Frequency
Leachate Extraction Blank (LEB)	For each SDG, an LEB for each extraction procedure.

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EXHIBIT D

TRACE CONCENTRATIONS OF
VOLATILE ORGANIC COMPOUNDS ANALYSIS

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Exhibit D – Trace Concentrations of
Volatile Organic Compounds Analysis

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Exhibit D – Trace Concentrations of
Volatile Organic Compounds Analysis

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water samples containing trace concentrations of the volatile analytes listed in the Target Analyte List (TAL) for trace volatiles in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. The majority of the samples are expected to be obtained from drinking water and well/groundwater type sources around Superfund sites. The method is based on the U.S. Environmental Protection Agency (EPA) Method 524.2. The sample preparation and analysis procedures included in this method are based on purge-and-trap (P/T) Gas Chromatograph/Mass Spectrometer (GC/MS) techniques.

1.2 Problems that have been associated with the following analytes using this method include:

- Chloromethane, vinyl chloride, bromomethane, and chloroethane may display peak broadening if the analytes are not delivered to the GC column in a tight band.
- Acetone, hexanone, 2-butanone, and 4-methyl-2-pentanone have poor purge efficiencies and may be lost if purge flow is too slow.
- 1,1,1-Trichloroethane and all of the dichloroethanes may dehydrohalogenate during storage or analysis.
- Tetrachloroethane and 1,1-dichloroethane may be degraded by contaminated transfer lines in P/T systems and/or active sites in trapping materials.
- Chloromethane and other gases may be lost if the purge flow is too fast.
- Bromoform is one of the analytes most likely to be adversely affected by cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by the tuning of 4-bromofluorobenzene (BFB) at ions m/z 174/176. Increasing the m/z 174/176 ratio within the specified Quality Control (QC) limits may improve bromoform response.
- Due to the lower quantitation limits required by this method, extra caution must be exercised when identifying compounds.

2.0 SUMMARY OF METHOD

2.1 Water

An inert gas is bubbled through a 25 milliliter (mL) sample contained in a specially designed purging chamber at ambient temperature. Higher purge temperatures may be used, provided that all technical acceptance criteria are met for all standards, samples, and blanks. The same purge conditions must be used for all associated standards, samples, and blanks. The purgeable compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeable compounds are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeable compounds onto a GC capillary column. The GC is temperature-programmed to separate the purgeable compounds, which are then detected with an MS.

2.2 Soil/Sediment

Not applicable to this method.

2.3 Wipes

Not applicable to this method.

2.4 Waste

Not applicable to this method.

2.5 Non-Target Compounds

Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library. Non-target compounds are quantitated by comparing the area response from the total Reconstructed Ion Chromatogram (RIC) for the non-target compound peaks to the area response produced by the nearest internal standard compound. A Relative Response Factor (RRF) of 1 is assumed.

3.0 DEFINITIONS

See Exhibit G – Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

4.1.1 Method interference may be caused by impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by analyzing laboratory method and instrument blanks as described in Section 12.1. The use of non-polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.1.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples must be stored separately from other laboratory samples and standards, and must be analyzed in a room whose atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis.

4.1.3 Contamination by carryover can occur whenever high-level and trace-level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank, or the next sample must be closely monitored to check for cross-contamination. 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 a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

4.1.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine the presence of methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all Gas Chromatography (GC) carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken. At the time of sample receipt, the Contractor must prepare a 40 mL VOA vial containing reagent water to be stored as a storage blank with each group of samples (Section 12.1.4).

4.2 Matrix Interferences

Matrix interferences may be caused by compounds that are purged or co-extracted from the sample. The extent of matrix interferences will vary considerably depending on the nature of the site being sampled.

5.0 SAFETY

See Section 12.0 of Exhibit D – Introduction to Organic Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 General Laboratory Equipment

6.1.1 Bottle - 15 mL, screw-cap, with PTFE cap liner.

6.1.2 Pasteur Pipettes - Disposable.

6.1.3 pH Paper - Wide range.

6.1.4 Syringes - 25 mL, gas-tight with shut-off valve.

6.1.5 Micro Syringes – 10 microliters (µL) and larger, 0.006 inch [0.15 millimeter (mm)] ID needle. All micro syringes shall be visually inspected and documented monthly.

6.1.6 Syringe Valve - Two-way, with Luer-Lok ends (three each), if applicable to the purging device.

6.1.7 Vials and Caps - Assorted sizes.

6.1.8 Volumetric Flasks - Class A with ground-glass stoppers.

6.2 Glassware/Extraction/Cleanup Equipment

Not applicable to this method.

6.3 Analytical Instrumentation

6.3.1 Gas Chromatograph

The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout desorption and temperature program operations. The system must include or be interfaced to a P/T system as specified in Section 6.3.4 and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants, or flow controllers with rubber components, are not to be used.

6.3.2 Gas Chromatography Columns

Recommended Column: Minimum length 30 meter (m) x 0.53 mm ID fused silica wide-bore capillary column with a 6% Cyanopropylphenyl 94% Dimethyl Polysiloxane phase having a 3 micrometer (µm) film thickness (i.e., VOCOL, Rtx®-502.2, DB-624, Rtx®-624, CP-Select 624CB, or equivalent fused silica wide-bore capillary column). A description of the GC column used for analysis shall be provided in the SDG Narrative. Packed GC columns cannot be used.

The column shall be able to accept up to 1000 nanograms (ng) of each analyte listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits without becoming overloaded.

6.3.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants that interfere with the identification and quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits.
- The analytical results generated using the column meet the initial calibration, initial calibration verification (ICV), and continuing calibration verification (CCV) technical acceptance criteria (Sections 9.3.5, 9.4.5, and 9.5.5) and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits. Sufficient chromatographic resolution is achieved when the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights.
- The column provides equal or better resolution of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits than the columns listed in Section 6.3.2.

6.3.2.1.1 As applicable, follow the manufacturer’s instructions for use of its product.

6.3.2.1.2 The Contractor must maintain documentation that the column met the criteria in Section 6.3.2.1. The minimum documentation is as follows:

6.3.2.1.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.3.2.1.2.2 RICs and data system reports generated on the GC/MS used for EPA Contract Laboratory Program (CLP) analyses:

- From instrument blanks that demonstrate that there are no contaminants that interfere with the volatile analysis when using the alternate column; and
- From initial calibration, ICV, and CCV standards analyzed using the alternate column.

6.3.2.1.3 Based on the Contractor-generated data described above, the Contractor shall complete a written comparison/review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Sections 9.3.5, 9.4.5, and 9.5.5;
- The low-point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high-point initial calibration standard analysis was not overloaded; and
- The column does not introduce contaminants that interfere with the identification and/or quantitation of analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits.

6.3.2.1.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request by the EPA Regional CLP Contracting Officer's Representative (COR).

6.3.3 Mass Spectrometer

The MS must be capable of scanning from 35-300 atomic mass units (u) every 2 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the BFB GC/MS performance check technical acceptance criteria in Table 2 – 4-Bromofluorobenzene Key Ions and Ion Abundance Criteria, when 50 ng of BFB is injected through the GC inlet. The instrument conditions required for the acquisition of the BFB mass spectrum are given in Section 9.2.4.

NOTE: To ensure sufficient precision of mass spectral data, the MS scan rate should allow acquisition of at least five spectra while a sample compound elutes from the GC. The P/T GC/MS system must be in a room whose atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. The instrument must be vented to the outside of the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.3.3.1 Gas Chromatograph/Mass Spectrometer Interface

Any GC/MS interface may be used that gives acceptable calibration points at 12.5 ng or less per injection for each of the purgeable non-ketone target analytes and Deuterated Monitoring Compounds (DMCs), and achieves all acceptable performance criteria. GC/MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.3.4 Purge-and-Trap Device

The P/T device consists of three separate pieces of equipment: the sample purge chamber, the trap, and the desorber. The analyst either manually or automatically (through an automated P/T device separate or integral with the GC) samples an appropriate volume (e.g., 25 mL) from the vial; adds DMCs, matrix spikes (MS), and internal standards to the sample; and transfers the sample to the purge device. The device also purges the volatile organic compounds (VOCs) using an inert gas stream and traps the released VOCs for subsequent desorption into the GC. Such systems shall meet the following specifications:

6.3.4.1 The sample purge chamber must be designed to accept 25 mL samples with a water column at least 10 centimeters (cm) deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

6.3.4.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inches (2.667 mm). The trap must be packed to contain (starting from the inlet) 0.5 cm silanized glass wool, and the following minimum lengths of adsorbent:

- 8 cm of 2,6-diphenylene oxide polymer (60/80 mesh chromatographic grade Tenax GC or equivalent).
- 1 cm methyl silicone packing, 3.0% OV-1 on Chromasorb W, 60/80 mesh (or equivalent).
- 8 cm of silica gel, 35/60 mesh (or equivalent).
- 7 cm of coconut charcoal.

6.3.4.3 Alternate sorbent traps may be used if:

- The trap packing materials do not introduce contaminants that interfere with identification and quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated using the trap meet the initial calibration, ICV, and CCV technical acceptance criteria listed in the analytical method and the CRQLs listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits; and
- The trap must be capable of accepting up to 1000 ng of each analyte listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits without becoming overloaded.

6.3.4.3.1 Before use of any trap other than the one specified in Section 6.3.4.2, the Contractor must first meet the criteria listed in Section 6.3.4.3. Once this has been demonstrated, the Contractor must document its use in each SDG Narrative by specifying the trap composition (packing material/brand name, amount of packing material). Other sorbent traps include, but are not limited to: Tenax/Silica Gel/Carbon Trap from EPA

Method 524.2, Tenax - GC/Graphpac-D Trap (Alltech) or equivalent, and Vocarb 4000 Trap (Supelco) or equivalent.

6.3.4.3.2 The Contractor must maintain documentation that the alternate trap meets the criteria listed in Section 6.3.4.3. The minimum documentation requirements are as follows:

6.3.4.3.2.1 Manufacturer-provided information concerning the performance characteristics of the trap.

6.3.4.3.2.2 RICs and data system reports generated on the Contractor's GC/MS used for CLP analyses:

- From instrument blank analyses that demonstrate that there are no contaminants that interfere with the volatile analysis when using the alternate trap; and
- From initial calibration, ICV, and CCV standards analyzed using the trap specified in Section 6.3.4.2.

6.3.4.3.2.3 Based on Contractor-generated data described above, the Contractor must complete a written comparison/review that has been signed by the Laboratory Manager certifying that:

- The alternate trap performance meets the technical acceptance criteria listed in Sections 9.3.5, 9.4.5, and 9.5.5;
- The low-point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high-point initial calibration standard analysis was not overloaded; and
- The alternate trap materials do not introduce contaminants that interfere with the identification and/or quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits.

6.3.4.3.2.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request of the EPA Regional CLP COR.

6.3.4.3.2.5 A description of the trap used for analysis shall be provided in the SDG Narrative.

6.3.4.4 The P/T apparatus may be assembled as a separate unit or be an integral unit coupled with a GC.

6.3.4.5 The desorber shall be capable of rapidly heating the trap to the desorb temperature recommended for the trap in use. The polymer section of the trap should not be heated higher than 180°C and the remaining sections should not exceed 220°C during bake-out mode.

6.4 Data Systems/Data Storage

A computer system must be interfaced to the MS that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be

available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows comparing sample spectra against reference library spectra. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

7.0 REAGENTS AND STANDARDS

The Contractor must provide all standards to be used with the contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Organic Analytical Methods, Section 11. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1 Reagents

7.1.1 Reagent Water - Reagent water is defined as water in which an interferent is not observed at or above the CRQL for each analyte of interest.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g [1 pound (lb)] of activated carbon.

7.1.1.2 Reagent water may also be generated using a water purification system.

7.1.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle, seal with a PTFE-lined septum, and cap.

7.1.2 Methanol - High Performance Liquid Chromatography (HPLC) quality or equivalent - Each lot of methanol used for analysis under the contract must be purged with nitrogen and must be demonstrated to be free of contaminants that interfere with the measurement of purgeable analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits.

7.2 Standards

7.2.1 Stock Standard Solutions

Stock standard solutions are defined as standards that are to be used to produce working standards. They may be in the form of single compounds or mixtures. They may be in methanol from pure standard materials or purchased as pre-made solutions. Prepare fresh stock standards every 6 months, or sooner if the standard has degraded or evaporated.

7.2.2 Working Standards

7.2.2.1 Initial and Continuing Calibration Solutions

Prepare working calibration standard solution(s) containing all of the purgeable target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits) in methanol. Prepare fresh calibration standard solution(s) every month, or sooner if the solution has degraded or evaporated.

NOTE: The Contractor may prepare a calibration standard containing all of the non-ketones and a separate standard containing ketones.

7.2.2.1.1 Add a sufficient amount of each working standard to a 25 mL aliquot of reagent water to produce the desired calibration standard concentrations listed in Section 7.2.2.1.2 or 7.2.2.1.4.

7.2.2.1.2 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target analytes and the DMCs at the following levels: all non-ketone target analytes and associated DMCs at 0.50, 1.0, 5.0, 10, and 20 µg/L (in Table 3 – Trace Volatile Deuterated Monitoring Compounds and the Associated Target Analytes); all ketones and their associated DMCs (see Table 3 - Trace Volatile Deuterated Monitoring Compounds and the Associated Target Analytes) at 5.0, 10, 50, 100, and 200 µg/L. All three xylene isomers (o-, m-, and p-xylene) must be present in the calibration standards. The o-xylene calibration standard concentrations must be at 0.50, 1.0, 5.0, 10, and 20 µg/L, while the concentration of the m- plus the p-xylene isomers must total 0.50, 1.0, 5.0, 10, and 20 µg/L.

7.2.2.1.3 Calibration standards must be prepared in a volumetric flask or in the syringe used to inject the standard into the purging device.

7.2.2.1.4 For CCV (opening and closing CCVs), the standard shall be at a concentration equivalent to the mid-level calibration standards: 5.0 µg/L for non-ketones and 50 µg/L for ketones.

7.2.2.1.5 The methanol contained in each of the aqueous calibration standards must not exceed 1% by volume.

7.2.2.2 Initial Calibration Verification Solution

Prepare the working initial calibration standard solution containing all of the purgeable target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits) from an alternate source or a different lot than that used for the initial calibration (ICAL) standard analyses in methanol. Prepare a fresh calibration standard solution every month, or sooner if the solution has degraded or evaporated.

7.2.2.2.1 The ICV standard shall be at a concentration equivalent to the mid-level calibration standards: 5.0 µg/L for non-ketones and 50 µg/L for ketones.

7.2.2.2.2 The ICV standard shall be prepared by the same procedures as the CCVs.

7.2.2.3 Instrument Performance Check Solution

Prepare the instrument performance check solution containing BFB in methanol. If the BFB solution is added to the mid-level calibration standard (5.0 µg/L for non-ketones and 50 µg/L for ketones), add a sufficient amount of BFB to result in a 2.0 µg/L concentration of BFB (50 ng on-column). The BFB must be analyzed using the same GC and MS analytical conditions as are used for the calibration analysis.

7.2.2.4 Deuterated Monitoring Compound Spiking Solution

7.2.2.4.1 Prepare a DMC spiking solution in methanol (or in deuterated methanol) containing the compounds listed in Table 3 - Trace Volatile Deuterated Monitoring Compounds and the Associated Target Analytes.

7.2.2.4.2 DMCs are to be added to each sample and blank, as well as initial calibration standards, ICV standard, and CCV standards.

7.2.2.4.3 For samples and blanks, add sufficient amount of the DMC spiking solution to each 25 mL of sample to result in 0.125 µg for each non-ketone DMC and 1.25 µg for each ketone DMC.

7.2.2.4.4 For ICAL, ICV, and CCV standards, add sufficient amounts of the DMC spiking solution to each 25 mL aliquot of calibration standard to result in the concentrations listed in Section 7.2.2.1.2 (initial calibration), Section 7.2.2.2.1 (ICV), and Section 7.2.2.1.4 (CCV).

7.2.2.4.5 Prepare a fresh DMC spiking solution every month, or sooner if the standard has degraded or concentrated.

7.2.2.5 Matrix Spiking Solution

If Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis is requested at the time of scheduling, prepare a spiking solution in methanol that contains the following analytes at a concentration of 12.5 µg/mL: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Prepare fresh spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.2.6 Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing 1,4-dichlorobenzene-d_4, chlorobenzene-d_5, and 1,4-difluorobenzene in methanol. Add a sufficient amount of the internal standard spiking solution to 25 mL of samples including MS/MSDs, blanks, and calibration standards to result in a 5.0 µg/L concentration or the addition of 0.125 µg for each internal standard. Prepare a fresh internal standard spiking solution every month, or sooner if the standard had degraded or evaporated.

7.2.3 Storage of Standard Solutions

7.2.3.1 Store the stock standards in PTFE-sealed screw-cap bottles with zero headspace at -10°C to -20°C.

7.2.3.2 Aqueous standards may be stored for up to 24 hours if held in PTFE-sealed screw-cap vials with zero headspace at ≤6°C, but not frozen. If not stored as such, the standards must be discarded after 1 hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be

kept up to 12 hours in purge tubes connected via the autosampler to the P/T device.

7.2.3.3 Standard solutions purchased from a chemical supply company as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor that are immediately ampulated in glass vials may be retained for 2 years from the preparation date. The expiration date of the ampulated standards, upon the breaking of the glass seal, is 6 months (or sooner if the standard has degraded or evaporated).

7.2.3.4 Protect all standards from light.

7.2.3.5 Purgeable standards must be stored separately from other standards, samples, and blanks.

7.2.3.6 The Contractor is responsible for maintaining and verifying the integrity of standard solutions prior to use. Storage of standard solutions in the freezer may cause some standards to precipitate. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution.

7.2.3.6.1 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases may need to be replaced after 1 month for working standards and 6 months for opened stocks, or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

7.2.4 Temperature Records for Storage of Standards

7.2.4.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators/freezers.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in glass containers having a total volume of at least 40 mL with a PTFE-lined septum and an open top screw-cap.

8.1.2 The containers should have been filled in such a manner that no air bubbles are entrained to create a headspace in the vial.

8.1.3 The samples are preserved to a pH ≤2 at the time of collection.

8.1.4 A total of three vials per field sample is the recommended amount the Contractor should receive.

NOTE: If MS/MSD analysis is required for a particular sample, two additional vials should be sent by the field samplers. Contact the Sample Management Office (SMO) if insufficient sample for MS/MSD analysis has been provided.

8.2 Procedure for Sample Storage

8.2.1 The samples must be protected from light and refrigerated at ≤6°C, but not frozen, from the time of receipt until 60 days after delivery of a complete, reconciled data package to the EPA.

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants and in a refrigerator used only for storage of volatile samples received under the contract.

8.2.3 All volatile samples in an SDG must be stored together in the same refrigerator.

8.2.4 Storage blanks shall be stored at ≤6°C, but not frozen, with samples within an SDG until all such samples are analyzed.

8.3 Contract Required Holding Times

Analysis of water samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Initial Instrument Set-up

9.1.1 Purge-and-Trap

9.1.1.1 The recommended P/T analytical conditions are provided in Table 5 – Purge-and-Trap Analytical Conditions. The conditions are suggested, but other conditions may be used, provided that all technical acceptance criteria are met for all standards, samples, and blanks:

9.1.1.2 Assemble a P/T device that meets the specifications in Section 6.3.4 and that is connected to a GC/MS system.

9.1.1.3 P/T instrumentation that allows internal standards and DMCs to be automatically added to each sample is widely available. Some of this instrumentation may be set-up by the manufacturer to add only 1 µL of internal standard or DMCs. The 1 µL addition of standards will be allowed if the addition is done solely in an automated manner, and if the final concentration of the following standards in the 25 mL water samples and blanks can be met: 5 µg/L for internal standards; the concentrations listed in Section 7.2.2.1.2 for DMCs in the initial calibration; the concentrations listed in Section 7.2.2.2.1 for DMCs in the ICV; and the concentrations listed in Section 7.2.2.1.4 for DMCs in the CCV.

9.1.1.4 Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 mL/minute flow of inert gas according to the manufacturer's recommendations. Do not vent the trap effluent onto the analytical column. Prior to daily use, condition the trap by heating at 180°C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be conditioned through the temperature program prior to the analysis of samples and blanks.

9.1.1.5 Optimize P/T conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same P/T conditions must be used for the analysis of all standards, samples, and blanks.

9.1.1.6 A moisture reduction/water management system may be used to improve the chromatographic performance by controlling moisture if:

- The system does not introduce contaminants that interfere with identification and quantitation of target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated when using the moisture reduction/water management system meet the initial calibration, initial calibration verification, and continuing calibration verification technical acceptance criteria listed in the analytical method and the CRQLs listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits;
- All calibration standards, samples, and blanks are analyzed under the same conditions; and

- The Contractor performs acceptably on the Performance Evaluation (PE) samples using this system.

9.1.2 Gas Chromatograph

9.1.2.1 The recommended GC analytical conditions are provided in Table 6 – Gas Chromatograph Analytical Conditions. The conditions are recommended unless otherwise noted. GC conditions must achieve all performance criteria required for initial calibration, initial calibration verification, and continuing calibration.

9.1.2.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSDs.

9.1.2.3 Target analytes that are isomers (e.g., dichlorobenzenes) must be at least 50% resolved from each other. For xylene isomers, the two peaks representing o-xylene, m- and p-xylene, respectively, must be at least 50% resolved.

9.1.2.4 If the gaseous analytes chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shape, are not separated from the solvent front, or are not resolved greater than 90.0% from each other, then a subambient oven controller must be used, and the initial temperature must be less than or equal to 10°C.

9.1.3 Mass Spectrometer

The recommended MS analytical conditions are provided in Table 7 – Mass Spectrometer Analytical Conditions.

9.2 Instrument Performance Check

9.2.1 Summary of GC/MS Instrument Performance Check

9.2.1.1 The GC/MS system must be tuned to meet the manufacturer’s specifications, using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.2.3).

9.2.1.2 Prior to the analysis of any samples, including MS/MSDs, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing BFB.

9.2.2 Frequency of GC/MS Instrument Performance Check

The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples, blanks, or standards are to be analyzed. The 12-hour period for the GC/MS instrument performance check, calibration standards (initial calibration, ICV, or CCV), blank, and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. However, in cases where a closing CCV can be used as an opening CCV for the next 12-hour period, then an additional BFB tune is not required, and the 12-hour period begins with the injection of the CCV. The time period ends after 12 hours have elapsed according to the system clock.

9.2.3 Procedure for GC/MS Instrument Performance Check

The analysis of the instrument performance check solution shall be performed as follows:

- As an injection of up to 50 ng of BFB into the GC/MS.
- By adding a sufficient amount of BFB solution (Section 7.2.2.3) to 25 mL of reagent water to result in a 2.0 µg/L concentration of BFB.
- By adding a sufficient amount of BFB solution to the mid-level calibration standard to result in a 2 µg/L concentration of BFB.

9.2.4 Technical Acceptance Criteria for GC/MS Instrument Performance Check

9.2.4.1 The GC/MS system must be tuned at the frequency described in Section 9.2.2.

9.2.4.2 The abundance criteria listed in Table 2 – 4-Bromofluorobenzene Key Ions and Ion Abundance Criteria, must be met for a 25 ng injection of BFB. The mass spectrum of BFB must be acquired in the following manner:

9.2.4.2.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

9.2.4.2.2 Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of BFB. Do not background subtract part of the BFB peak.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a BFB analysis must be analyzed under identical GC/MS instrument analytical conditions.

9.2.5 Corrective Action for GC/MS Instrument Performance Check

9.2.5.1 If the BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source or take other corrective actions to achieve the technical acceptance criteria.

9.2.5.2 Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples (including MS/MSDs) and required blanks, and after the instrument performance check technical acceptance criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations (Section 7.2.2.1.2) to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target analytes and DMCs.

9.3.2 Frequency of Initial Calibration

9.3.2.1 Each GC/MS system must be calibrated prior to analyzing samples, whenever the Contractor takes corrective action that may change or affect the initial calibration criteria (e.g., ion source cleaning or repair, column replacement, etc.), or if the CCV technical acceptance criteria have not been met.

9.3.2.2 If time remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed (Section 9.3.5). It is not necessary to analyze another CCV standard. A method blank is required.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set up the GC/MS system as described in Section 9.1.

9.3.3.2 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.3.3.3 Add sufficient amount of the internal standard solution (Section 7.2.2.6) to each of the five aqueous calibration standard solutions (Section 7.2.2.1.2) containing the DMCs (Section 7.2.2.4.1) at the time of purge. Analyze each calibration standard according to Section 10.0 and outlined in Section 9.3.1. The initial calibration sequence is listed below.

INITIAL CALIBRATION SEQUENCE

1. GC/MS Instrument Performance Check
2. CS1 Initial Calibration Standard
3. CS2 Initial Calibration Standard
4. CS3 Initial Calibration Standard
5. CS4 Initial Calibration Standard
6. CS5 Initial Calibration Standard

9.3.4 Calculations for Initial Calibration

9.3.4.1 Calculate the RRF for each purgeable target analyte and DMC using Equation 1. The primary characteristic ions used for quantitation are listed in Table 8 – Characteristic Ions for Trace Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards. If an interference prevents the use of a primary ion for a given internal standard, use a secondary ion listed in the same table. Assign the target analytes and DMCs to an internal standard according to Table 9 – Trace Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

NOTE: Unless otherwise stated, the area response of the primary characteristic ion is the quantitation ion.

EQ. 1 Relative Response Factor

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

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (Table 8 – Characteristic Ions for Trace Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (Table 8 – Characteristic Ions for Trace Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards). The target analytes are listed with their associated internal standards in Table 9 - Trace Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

C_{is} = Concentration or amount of the internal standard

C_x = Concentration or amount of the analyte to be measured

9.3.4.2 Calculating the RRFs of the xylenes requires special attention. Report an RRF for m,p-xylene and one for o-xylene. On the available capillary columns, the m,p-xylene isomers coelute. Therefore, when calculating the RRF in the equation above, use the area response (A_x) and concentration (C_x) of the peak from o-xylene, and A_X and C_X of the peak from the m,p-xylene isomers respectively.

9.3.4.3 The Mean ($\overline{RRF}$) must be calculated for all analytes according to Equation 2.

9.3.4.4 Calculate the Percent Relative Standard Deviation (%RSD) of the RRF values for each purgeable target analyte and DMC over the initial calibration range using Equation 3 in conjunction with Equations 2 and 4.

9.3.4.4.1 Equation 2 is the general formula for the mean of a set of values.

EQ. 2 Mean Value

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

WHERE,

X_i = Value

$\overline{X}$ = Mean value

n = Number of values

9.3.4.4.2 Equation 3 is the general formula for the relative standard deviation.

EQ. 3 Percent Relative Standard Deviation

$$\%RSD = \frac{SD_{RRF}}{\overline{X}} \times 100$$

WHERE,

SD_{RRF} = Standard deviation of initial calibration RRFs (per compound) from EQ. 4

$\overline{X}$ = Mean value of the initial calibration RRFs (per compound)

9.3.4.4.3 Equation 4 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 4 Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

WHERE,

X_i = Each individual value used to calculate the mean

$\overline{X}$ = The mean of n values

n = Total number of values

9.3.5 Technical Acceptance Criteria for Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentrations described in Section 7.2.2.1.2, and at the frequency described in Section 9.3.2 on a GC/MS system meeting the BFB technical acceptance criteria (Section 9.2.4).

9.3.5.2 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.3.5.3 The RRF at each calibration concentration for each target analyte and DMC that has a required minimum RRF value must be greater than or equal to the compound's minimum RRF listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds.

9.3.5.4 The %RSD for each target analyte or DMC listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds must be less than or equal to the value listed.

9.3.5.5 Up to two target analytes and DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds.

9.3.5.6 Up to two target analytes and DMCs (excluding those with maximum %RSD requirements of 40.0%) may fail to meet the criteria listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds, but these compounds must still meet the maximum %RSD requirements of 40.0%.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, service the P/T device, or take other corrective actions to achieve the technical acceptance criteria.

9.3.6.2 It may be necessary to adjust the purge gas (helium) flow rate (normally in the range of 25-40 mL/minute). Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

9.3.6.3 Any samples or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.4 Initial Calibration Verification

9.4.1 Summary of Initial Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check and initial calibration technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an ICV (containing all the purgeable target analytes from an alternate source or a different lot than the ICAL standards, and the DMCs and internal standards from the same source or lot as in the ICAL standards) to ensure that the instrument is calibrated accurately.

9.4.2 Frequency of Initial Calibration Verification

The calibration for each GC/MS system used for analysis must be verified with an ICV at the frequency of one per ICAL analytical sequence. The ICV shall be analyzed following that last ICAL standard analysis and prior to any method blank, sample, or applicable CCV analysis.

Injection #	**Material Injected**
1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	BFB then CS1–CS5 First 6 steps of the initial calibration
7th - ICV	ICV
8th - blanks, samples, MS/MSDs	Blanks, samples, and MS/MSDs
9th - Subsequent Samples	

9.4.3 Procedure for Initial Calibration Verification

9.4.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.4.3.2 Add sufficient amount of internal standard solution (Section 7.2.2.6) to the ICV (Section 7.2.2.2) and the DMC solution (Section 7.2.2.4). Analyze the ICV Standard according to Section 10.0.

9.4.4 Calculations for Initial Calibration Verification

9.4.4.1 Calculate an RRF for each target analyte and DMC according to Section 9.3.4.1.

9.4.4.2 Calculate the Percent Difference (%D) between the ICV RRF_c and the preceding initial calibration $\overline{RRF_i}$ for each purgeable target analyte and DMC using the following equation:

EQ. 5 Initial Calibration Verification Percent Difference

$$\%D = \frac{RRF_C - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

WHERE,

RRF_c = Relative Response Factor from current ICV standard

$\overline{RRF_i}$ = Mean Relative Response Factor from the preceding initial calibration

9.4.5 Technical Acceptance Criteria for Initial Calibration Verification

9.4.5.1 The concentration of the trace volatile organic target analytes and DMCs in the ICV must be at or near the mid-point concentration of the calibration standards (5.0 µg/L for non-ketones and 50 µg/L for ketones). The ICV must be analyzed at the frequency described in Section 9.4.2, on a GC/MS system meeting the BFB (Section 9.2.4) and the initial calibration (Section 9.3.5) technical acceptance criteria.

9.4.5.2 For an ICV, the RRF for each target analyte and DMC must be greater than, or equal to, the compound's minimum RRF listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds. Up to two target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds, but these compounds must still meet the minimum RRF requirements of 0.010.

9.4.5.3 For an ICV, the %D for each target analyte and DMC listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds must be in the inclusive range of the compound's %D values listed.

9.4.5.4 No quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Initial Calibration Verification

9.4.6.1 If the ICV technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3.

9.4.6.2 If the ICV fails to meet the technical acceptance criteria and a subsequent reanalysis of the ICV meets the technical acceptance criteria, proceed to the blank and sample analyses. All sample and required blank analyses must be associated to a compliant ICV analysis following the associated ICAL.

9.5 Continuing Calibration Verification

9.5.1 Summary of Continuing Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check, initial calibration, and ICV technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an opening CCV (containing all the purgeable target analytes, DMCs, and internal standards) to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the analytical method. A closing CCV using the same standard conditions as for the opening CCV is required after all samples and blanks have been analyzed, and before the end of the 12-hour period (refer to the analytical sequence provided in Section 9.5.2.3).

9.5.2 Frequency of Continuing Calibration Verification

9.5.2.1 The calibration for each GC/MS system used for analysis must be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the injection of BFB,

followed by the injection of the opening CCV solution. BFB may be added to the CCV solution, in which case only one injection is necessary. If a closing CCV meets the technical acceptance criteria for an opening CCV (Section 9.5.5) and samples are analyzed within that subsequent 12-hour period, then an additional BFB tune is not required and the 12-hour period begins with that calibration verification. If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then a BFB tune, followed by an opening CCV, is required and the next 12-hour period begins with the BFB tune (Section 9.2.2).

9.5.2.2 If time remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration and ICV, samples may be analyzed. A method blank is required.

9.5.2.3 After the injection of all samples and required blanks, and before the end of the 12-hour period, another injection of the CCV solution is required (closing CCV). The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence, provided that all technical acceptance criteria are met for an opening CCV in Section 9.5.5.

Time	**Injection #**	**Material Injected**
0 hr	1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	BFB then CS1–CS5 First 6 steps of the initial calibration
	7th - ICV	ICV
	8th - blanks, samples, MS/MSDs	Blanks, samples, and MS/MSDs
	9th – Subsequent Samples	
End 12 hr	Closing CCV (meeting Closing CCV criteria, but not Opening CCV)	CS3 – Closing CCV
New 12 hr	1st GC/MS Instrument Performance Check	BFB Instrument Performance Check
	2nd - Analysis past 12 hours Opening CCV	CS3 - Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
End 12 hr	Closing CCV (meeting Closing CCV criteria but not Opening CCV)	CS3 – Closing CCV
New 12 hr	1st Analysis Instrument Performance Check	BFB Instrument Performance Check
	2nd Analysis Opening CCV	CS3 – Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
		Storage Blank if previous sample is the last sample in SDG

Time	Injection #	Material Injected
End of 12 hr beginning of next 12 hr	Closing CCV (meeting Opening CCV criteria) Instrument Performance Check not required	CS3 – Closing CCV meeting Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
		Storage Blank (after last sample in SDG)
End of 12 hr	Closing CCV meeting criteria	CS3 – Closing CCV meeting Opening CCV

9.5.3 Procedure for Continuing Calibration Verification

9.5.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.5.3.2 Add sufficient amount of internal standard solution (Section 7.2.2.6) to the CCV (Section 7.2.2.1.4) and the DMC solution (Section 7.2.2.4). Analyze the CCV Standard according to Section 10.0.

9.5.4 Calculations for Continuing Calibration Verification

9.5.4.1 Calculate an RRF for each target analyte and DMC according to Section 9.3.4.1.

9.5.4.2 Calculate the %D between the CCV RRF_c and the most recent initial calibration $\overline{RRF_i}$ for each purgeable target analyte and DMC using the following equation:

EQ. 6 Internal Standard Calibration Percent Difference

$$\%D = \frac{RRF_C - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

WHERE,

RRF_c = Relative Response Factor from current CCV standard

$\overline{RRF_i}$ = Mean Relative Response Factor from the most recent initial calibration

9.5.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.5.5.1 The concentration of the trace volatile organic target analytes and DMCs in the opening and closing CCV must be at or near the mid-point concentration of the calibration standards (5.0 µg/L for non-ketones and 50 µg/L for ketones). The opening and closing CCV must be analyzed at the frequency described in Section 9.5.2, on a GC/MS system meeting the BFB (Section 9.2.4), the initial calibration (Section 9.3.5), and the ICV (Section 9.4.5) technical acceptance criteria.

9.5.5.2 For an opening or closing CCV, the RRF for each target analyte and DMC must be greater than, or equal to, the compound's minimum RRF listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds.

9.5.5.3 For an opening CCV, the %D for each target analyte and DMC listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Trace Volatile Organic Compounds must be in the inclusive range of the compound's %D values listed. For a closing CCV, the %D for each target analyte and DMC must be in the inclusive range of the compound's %D values listed. Up to two target analytes and/or DMCs in the closing CCV are allowed to exceed the %D values listed.

9.5.5.4 For an opening or closing CCV, up to two target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Section 9.5.5.2, but these compounds must still meet the minimum RRF requirements of 0.010.

9.5.5.5 No quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.5.6 Corrective Action for Continuing Calibration Verification

9.5.6.1 If the opening CCV technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3. If the closing CCV technical acceptance criteria are not met, then all samples and blanks analyzed within that 12-hour period must be reanalyzed at no additional cost to the EPA.

9.5.6.2 The Contractor shall follow the procedure in Section 10.2.12.1 if it cannot meet the control criteria after the analysis of an original undiluted or minimally diluted sample due to matrix interference. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the CCV technical acceptance criteria.

9.5.6.3 Any samples or required blanks analyzed when opening CCV technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.5.6.4 The corrective action for sample reanalysis is not required when noncompliant analytes or associated DMCs, in the opening or closing CCVs bracketing a dilution or a reanalysis, are not the same analytes or associated DMCs for which the dilution analysis or reanalysis was intended.

10.0 PROCEDURE

10.1 Introduction to Sample Analysis

10.1.1 Samples shall be analyzed only after the GC/MS system has met the technical requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration. All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

NOTE: Contact SMO if sample vials have bubbles entrained resulting in headspace.

10.2 Procedure for Sample Analysis

10.2.1 If time remains in the 12-hour period (as described in Section 9.2.2), samples may be analyzed without analysis of a CCV standard.

10.2.2 If the autosampler can automatically sample the appropriate volume, then Sections 10.2.3 - 10.2.5 are performed by the autosampler.

10.2.3 Remove the plunger from a 25 mL syringe and attach a closed syringe valve. Open the sample or standard container that has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Invert the syringe, open the syringe valve, and vent any residual air while adjusting the sample volume to 25 mL.

10.2.4 This process of taking an aliquot destroys the validity of the sample for future analysis, unless the excess sample is immediately transferred to a smaller vial with zero headspace and stored at ≤6°C, but not frozen. Therefore, if only one sample vial is provided, the analyst must fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly. Filling one 25 mL syringe would allow only one analysis of that sample. If an analysis is needed from the second 25 mL syringe, it must be performed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

10.2.5 Add a sufficient amount of the DMC spiking solution (Section 7.2.2.4.1) and a sufficient amount of internal standard spiking solution (Section 7.2.2.6) through the valve bore of the syringe, then close the valve. Invert the syringe 3 times. The DMCs and internal standards may be mixed and added as a single spiking solution.

10.2.6 Once the sample aliquots have been taken from the VOA vial, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do **not** add pH paper to the vials). Record the pH of each sample and report these data in the SDG Narrative, following the instructions in Exhibit B – Reporting and Deliverables Requirements. No pH adjustment is to be performed by the Contractor.

10.2.7 Attach the valve assembly on the syringe to the valve on the sample sparger. Open the valves and inject the sample into the purging chamber.

10.2.8 Close both valves and purge the sample under the same conditions as the initial calibration.

10.2.9 Sample Desorption - After the purge is complete, attach the trap to the GC, adjust the P/T system to the desorb mode, initiate the temperature program sequence of the GC, and start data acquisition. Introduce the trapped material into the GC column by rapidly heating the trap to the appropriate desorb temperature while backflushing the trap with inert gas. While the trapped material is being introduced into the GC, empty the sample sparger and rinse it with reagent water. 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 sample sparger with a detergent solution, rinse it with reagent water, and then dry it in an oven at 105°C.

10.2.10 Trap Reconditioning - After desorbing the sample, recondition the trap in accordance with manufacturer's instructions with the recommended trap recondition for a minimum of 7.0 (±0.1) minutes at 180°C. The same conditions must be used for all analyses.

10.2.11 Termination of Data Acquisition - 3 minutes after all the purgeable target analytes have eluted from the GC, terminate the MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and appropriate EICPs.

10.2.12 Sample Dilutions

10.2.12.1 The Contractor shall analyze samples undiluted, or at minimal dilution. Samples may be diluted because of target analyte concentration exceeding the concentration of the same target analyte in the high standard, or because of excessive matrix interference that hinders accurate quantitation. It is highly recommended that screening analysis be performed prior to sample analysis to determine estimated compound concentration and matrix problems.

10.2.12.2 In the event that interference precludes accurate quantitation using the primary quantitation ion, but a secondary ion with less interference could be used instead, then secondary ion quantitation should be considered (see Section 11.2.1.4).

10.2.12.3 Use the results of the original sample analysis to determine the approximate Dilution Factor (DF) required to get the highest concentration of the analyte within the calibration range.

10.2.12.4 The DF chosen must keep the concentrations of the trace volatile target analytes that required dilution within the upper half of the initial calibration range.

10.2.12.5 If a sample requires a DF of twenty or greater to meet the criteria in Section 10.2.12.3, then the Contractor shall contact SMO. SMO will in turn contact the EPA Region to determine whether the sample should be analyzed at low level. The results of all original trace level analyses shall also be reported.

10.2.12.6 All dilutions must be made just prior to GC/MS analysis of the sample. Until the diluted sample is in a gas-tight syringe, all steps in the dilution procedure must be performed without delay.

10.2.12.7 Samples may be diluted in a volumetric flask or in a 25 mL Luer-Lok syringe.

10.2.12.8 To dilute the sample in a volumetric flask, use the following procedure:

10.2.12.8.1 Select the volumetric flask that will allow for necessary dilution (25-100 mL).

10.2.12.8.2 Calculate the approximate volume of appropriately acidified reagent water that will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

10.2.12.8.3 Inject the proper sample aliquot from a syringe into the volumetric flask. Only aliquots of 1 mL increments are permitted. Dilute the aliquot to the mark on the flask with reagent water. Cap the flask and invert it 3 times.

10.2.12.8.4 Fill a 25 mL syringe with the diluted sample and analyze according to Section 10.2.

10.2.12.9 To dilute the sample in a 25 mL syringe, use the following procedure:

10.2.12.9.1 Calculate the volume of the reagent water necessary for the dilution. The final volume of the diluted sample should be 25 mL.

10.2.12.9.2 Close the syringe valve, remove the plunger from the syringe barrel, and pour reagent water into the syringe barrel to just short of overflowing.

10.2.12.9.3 Replace the syringe plunger and compress the water.

10.2.12.9.4 Invert the syringe, open the syringe valve, and vent any residual air. Adjust the water volume to the desired amount.

10.2.12.9.5 Adjust the plunger to the 25 mL mark to accommodate the sample aliquot. Inject the proper aliquot of sample from another syringe through the valve bore of the 25 mL syringe. Close the valve and invert the syringe 3 times. Analyze according to Section 10.2.

10.2.12.10 All sample quality control criteria must be met for all diluted and undiluted sample analyses. Sample analyses that fail to meet the sample quality control criteria must be reanalyzed at no additional cost to the EPA.

10.2.12.11 If more than two analyses (i.e., from the original sample and more than one dilution, or from the most concentrated dilution analyzed and further dilutions) are required to get concentrations of all target analytes within the calibration range, contact SMO.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Analytes

11.1.1.1 The analytes listed in the TAL in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of the standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component within the Gas Chromatographic (GC) Relative Retention Time (RRT) unit window established from the 12-hour calibration standard; and
- Correspondence of the sample component and calibration standard analyte mass spectra.

11.1.1.2 For establishing correspondence of the GC RRT, the sample component RRT must be within ±0.06 RRT units of the RRT of the corresponding continuing calibration standard component. For reference, the standard must be analyzed in the same 12-hour period as the sample. If samples are analyzed during the same 12-hour period as the initial calibration standards, use the RRT values from the 5 µg/L standard. Otherwise, use the corresponding opening CCV standard. If coelution of interfering compounds prohibits accurate assignment of the sample component RRT from the total ion chromatogram, then the RRT should be assigned using the EICP for ions unique to the component of interest.

11.1.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS (as opposed to library spectra) are required. Once obtained, these standard spectra may be used for identification purposes, only if the Contractor's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the standard analysis that was also used to obtain the RRTs.

11.1.1.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:

11.1.1.4.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.1.1.4.2 The relative intensities of ions specified in the section above must agree within ±20% between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30-70%).

11.1.1.4.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra.

11.1.1.4.4 If an analyte cannot be verified by all of the spectral identification criteria listed in Section 11.1.1.4, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantitation and document in the SDG Narrative.

11.1.2 Identification of Non-Target Compounds

11.1.2.1 A library search shall be executed for non-target compounds for the purpose of tentative identification. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library, shall be used as the reference library.

11.1.2.2 All organic compounds that have not been positively identified as volatile target analytes using the procedures detailed in Section 11.1.1, or that are not DMCs, internal standards, or semivolatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, shall be tentatively identified via a forward search of NIST, Wiley, or equivalent mass spectral library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer-generated library search routines must not use normalizations which would misrepresent the library or unknown spectra when compared to each other.

11.1.2.3 Up to 30 non-alkane Tentatively Identified Compounds (TICs) of greatest apparent concentration shall be reported on Form 1B-OR. Peaks that are tentatively identified as straight-chain, branched, or cyclic alkanes, and are alone or part of an alkane series, shall be reported as "total alkanes". An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The concentrations of each of the alkanes is to be summed and reported as a single result for the "total alkanes". The alkanes are not to be counted as part of the 30 compounds individually reported as TICs on Form 1B-OR. Carbon dioxide and compounds with responses less than 10% of the internal standard with which they are to be quantified (as determined by inspection of the peak areas or height) are not to be reported (nor are they to be counted as part of the 30 compounds that are to be reported).

11.1.2.4 Rules for Making Tentative Identification

11.1.2.4.1 For compounds to be reported, as per the instructions in Section 11.1.2, identification (as generated by the library search program) of those receiving a library search match of 85% or higher should be considered a "probable match". The compound should be reported with the identification generated by the search program, unless the mass spectral interpretation specialist feels there is just evidence not to report the compound as identified by the library search program.

11.1.2.4.2 If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match. Do not report DMCs, internal standards, or analytes that are on the volatile

or semivolatile TAL, unless the semivolatile analysis is not being done.

11.1.2.4.3 If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethylnaphthalenes), the compound with the highest library search percent match should be reported (or first compound if library search matches are the same).

11.1.2.4.4 If the mass spectral interpretation specialist has just evidence to support reporting a compound with a tentative identification of something other than that generated by the library search program (with a library search result of 85% or greater), the laboratory shall include in the SDG Narrative the justification for not reporting a compound as listed by the search program. This narrative shall detail explicitly why a library search generated identification for a compound was rejected. If a TIC has obvious isomer analogs, the laboratory shall include in the SDG Narrative a statement indicating that the exact isomer configuration, as reported, may not be absolutely accurate.

11.1.2.4.5 If the library search produces no matches at or above 85%, the mass spectral interpretation specialists are encouraged to make a valid tentative identification of the compound. If no valid tentative identification can be made, the compound should be reported as "unknown". The mass spectral interpretation specialist should give additional classification of the unknown, if possible (e.g., "unknown aromatic compound", "unknown chlorinated compound", etc.).

11.1.2.4.6 The Chemical Abstracts Service (CAS) registry number is the unique identifier for each chemical compound. As the rules of chemical nomenclature have changed over time, each chemical substance is liable to have several names or synonyms: trade or brand name(s); generic or common name(s); trivial or systematic; or International Union of Pure and Applied Chemistry (IUPAC) name(s). Whether synonyms or other names are created for this compound, the CAS registry number will generally remain unchanged. The CAS registry number is simply an identifier which has no structural significance. Regardless of retention times (RTs), if the library search produces two or more compounds at or above 85% with the same Chemical Abstract Number, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match.

11.1.2.4.7 If the library search produces only one and the same compound (i.e., the same CAS registry number) with the match at or above 85% at two different RTs, the compound having the highest percent match should be reported as TIC and the other one could be reported as unknown. If both TICs have the same percent match for the same compound, one of the TICs could be reported as unknown. Such justifications should be included in the SDG Narrative.

11.2 Quantitative Analysis

11.2.1 Data Processing Procedure

11.2.1.1 Target analytes identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (Table 9 – Trace Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation). The EICP area of primary characteristic ions of analytes listed in Table 8 – Characteristic Ions for Trace Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards, are used for quantitation.

11.2.1.2 Xylenes are to be reported as "m,p-xylene" and "o-xylene". Because m- and p-xylene isomers co-elute, special attention must be given to the quantitation of the xylenes. In quantitating sample concentrations, be sure to use the correct corresponding RRF values.

NOTE: The area of each peak (i.e., the peaks for o-xylene and m,p-xylene) must appear on the quantitation report.

11.2.1.3 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene are to be reported separately.

11.2.1.4 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary quantitation ion, not when saturation occurs. If secondary ion quantitation is used, calculate an RRF using the area response (EICP) from the most intense secondary ion which is free of sample interferences, and document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

11.2.1.5 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitation. This normally occurs when there is compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific target analyte, DMC, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall also not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instances of manual integration must be documented in the SDG Narrative.

11.2.1.6 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This

applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

11.2.2 Target Analyte Calculations

11.2.2.1 Identified target analytes shall be quantified by the internal standard method using Equation 7. The internal standard used shall be that which is assigned in Table 9 - Trace Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation. The $\overline{RRF}$ from the initial calibration standard is used to calculate the concentration in the sample.

11.2.2.2 EQ. 7 Water Concentration

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_{is})(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and DMCs are listed in Table 8 – Characteristic Ions for Trace Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes are listed with their associated internal standards in Table 9 – Trace Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

I_{is} = Amount of internal standard added, in ng

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration standard

V_o = Total volume of water purged, in mL

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. For example, if 5.0 mL of sample is diluted to 25 mL with reagent water and purged, DF = 25 mL/5.0 mL = 5.0. If no dilution is performed, DF = 1.0.

11.2.3 Non-Target Compounds

11.2.3.1 An estimated concentration for TICs shall be determined by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used.

11.2.3.2 Equation 7 is also used for calculating TIC concentrations. Total area counts (or peak heights) from the total RICs are to be used for both the TIC to be measured (A_x) and the internal standard (A_{is}). An $\overline{RRF}$ of 1.0 is to be assumed.

11.2.4 Contract Required Quantitation Limit Calculation

EQ. 8 Water Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{V_c}{V_o} \times DF$$

WHERE,

Contract CRQL = CRQL value reported in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Required Contract Required Quantitation Limits

V_o, DF = As given in EQ. 7

V_c = Method required purge volume

11.2.5 Deuterated Monitoring Compound Recoveries

11.2.5.1 Calculate the concentration of each DMC using the same equation as used for target analytes (Equation 7).

11.2.5.2 Calculate the recovery of each DMC in all samples and blanks using Equation 9. Report the recoveries on the appropriate forms.

EQ. 9 DMC Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

WHERE,

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

11.3 Technical Acceptance Criteria for Sample Analysis

11.3.1 The sample must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, CCV, and blank technical acceptance criteria.

11.3.2 The sample and any required dilution must be analyzed within the contract required holding time.

11.3.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.3.4 The Percent Recovery (%R) of each of the DMCs in the sample must be within the recovery limits in Table 10 – Deuterated Monitoring Compound Recovery Limits. Up to three DMCs per sample may fail to meet the recovery limits listed in Table 10 – Deuterated Monitoring Compound Recovery Limits.

11.3.5 The EICP area for each of the internal standards in the sample must be within the range of 50-200% of its response in the most recent opening CCV standard analysis.

11.3.6 The RT shift for each of the internal standards in the sample must be within ±10 seconds of its RT in the most recent opening CCV standard analysis.

11.3.7 Excluding those ions in the solvent front, no ion may saturate the detector. No target analyte concentration may exceed the upper limit of the initial calibration range, unless a more diluted aliquot of the sample is also analyzed according to the procedures in Section 10.2.12.

11.3.8 The Contractor must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted. After a sample that contains a target analyte at a level exceeding the initial calibration range, or a non-target compound at

a concentration greater than 100 µg/L, or saturated ions from a compound (excluding the compound peaks in the solvent front), the Contractor must either:

- Analyze an instrument blank immediately after the contaminated sample. If an autosampler is used, an instrument blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The instrument blanks must meet the technical acceptance criteria for blank analysis (Section 12.1.3.5); or
- Monitor the sample analyzed immediately after the contaminated sample for the analytes that were in the contaminated sample and that exceeded the calibration range. The maximum carryover criteria are as follows: the sample must not contain a concentration above the CRQL for the target analytes, or above 2 µg/L for the non-target compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample must also meet the maximum contamination criteria.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or any samples not meeting the sample technical acceptance criteria will require reanalysis at no additional cost to the EPA.

11.4.2 Corrective actions for failure to meet technical acceptance criteria for instrument performance checks, initial calibration, ICV, CCV, and method blanks must be completed before the analysis of samples.

11.4.3 If the technical acceptance criteria for any of the internal standards and DMCs are not met, check calculations, internal standard and DMC spiking solutions, and instrument performance. It may be necessary to bake out the system to remove the water from the P/T transfer lines, to recalibrate the instrument, or take other corrective action procedures to meet the technical acceptance criteria.

11.4.4 After completing the corrective actions outlined above, the Contractor shall proceed to reanalyzing the sample as appropriate.

11.4.4.1 If the DMC recoveries do not meet the acceptance criteria in the initial sample analysis, reanalyze the sample.

- If the DMC recoveries fail to meet the acceptance criteria in the reanalyzed sample, then submit the data from both analyses. Distinguish between the initial analysis and the reanalysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.2 If the internal standard compound responses do not meet the acceptance criteria in the initial sample analysis, reanalyze the sample.

- If the internal standard compound responses are still noncompliant after the reanalysis, the Contractor shall dilute the original sample by a factor of 2-10 and reanalyze the sample. If the internal standard compound responses are acceptable in any of the subsequent diluted analyses, submit

the data from both the reanalysis and the compliant diluted analysis.

- If the internal standard compound responses fail to meet the acceptance criteria in the reanalysis and the subsequent diluted analysis, submit the data from both analysis. Distinguish between the initial analysis, reanalysis, and the diluted analysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.3 If the DMC recoveries in Section 11.4.4.1, the internal standard compound responses in Section 11.4.4.2, or both the DMC recoveries and the internal standard compound responses meet the acceptance criteria in the reanalyzed sample, it indicates that the problem was within the Contractor's control. Therefore, only submit the data from the reanalysis.

11.4.4.4 If the DMC recoveries or internal standard compound responses in a sample used for the MS/MSD analyses are outside the acceptance criteria, the Contractor shall proceed to the following corrective actions:

- If the DMC recoveries in a sample used for the MS/MSD analyses are outside the acceptance criteria, then the sample shall be reanalyzed only if the DMC recoveries meet the acceptance criteria in both the MS and MSD analyses.

- If the internal standard compound responses do not meet the acceptance criteria, the Contractor shall proceed to the reanalysis in Sections 11.4.4.2 and 11.4.4.3 even if the internal standard compound responses meet the technical acceptance criteria in the MS/MSD analyses.

11.4.5 If the Contractor needs to analyze more than one sample dilution other than the original analysis to have all concentrations of the target analytes within the initial calibration range, contact SMO. SMO will contact the EPA Region for instruction.

11.4.6 All samples to be reported to the EPA must meet the maximum carryover criteria in Section 11.3.8. If any sample fails to meet these criteria, each subsequent analysis must be checked for cross-contamination. The analytical system is considered contaminated until a sample has been analyzed that meets the maximum carryover criteria or an instrument blank has been analyzed that meets the technical acceptance criteria for blanks. If an instrument blank is not analyzed between consecutive samples that have the same analyte with a concentration exceeding the calibration range, then the second sample must be appropriately diluted as indicated in Section 10.2.12 and analyzed. If this analyte in the diluted analysis is detected at or below the adjusted CRQL, then all samples analyzed after the second sample that fail to meet maximum carryover criteria must be reanalyzed. If this analyte in the diluted analysis is detected within the calibration range, then no further corrective action is required.

11.4.7 Corrective Action for Internal Standard Compound Retention Times Outside Acceptance Criteria

11.4.7.1 If the internal standard compound RTs are not within their acceptance criteria, check the instrument for malfunctions. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample. If the instrument malfunction affected the

calibration, recalibrate the instrument before reanalyzing the samples.

11.4.7.2 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Reanalyze the sample. EXCEPTION: If the internal standard compound RTs in a sample used for an MS or MSD were outside the acceptance criteria, then it should be reanalyzed only if the internal standard compound RTs were within the acceptance criteria in both the MS/MSD analyses.
- If the internal standard compound RTs are within the acceptance criteria, then the problem was within the Contractor's control. Therefore, submit only data from the reanalysis when the internal standard compound RTs are within the acceptance limits.
- If the internal standard compound RTs are outside the acceptance criteria in the reanalysis, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables, using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.8 If the required corrective actions for sample reanalysis and/or dilution cannot be performed due to insufficient sample volume, the Contractor shall contact SMO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Summary

There are three different types of blanks required by this method: the method blank, the instrument blank, and the storage blank.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is a 25 mL aliquot of reagent water spiked with internal standard spiking solution (Section 7.2.2.6) and DMC solution (Section 7.2.2.4.1), and carried through the entire analytical procedure. The volume of the reference matrix must be approximately equal to the volume of samples associated with the blank. The purpose of the method blank is to determine the levels of contamination associated with processing and analysis of samples.

12.1.2.2 Frequency of Method Blank

12.1.2.2.1 The method blank must be analyzed at least once during every 12-hour period on each GC/MS system used for trace volatile analysis (See Section 9.2.2 for the definition of the 12-hour period).

12.1.2.2.2 The method blank must be analyzed after the initial calibration sequence (Section 9.3.1) if samples are analyzed before the 12-hour period expires. The method blank must be analyzed after the opening CCV and before any samples, including MS/MSDs, dilutions, or storage blanks are analyzed. A method blank must be analyzed in each 12-hour period in

which samples, including dilutions, MS/MSDs, and storage blanks from an SDG are analyzed.

12.1.2.3 Procedure for Method Blank

12.1.2.3.1 Method blanks shall be analyzed in the same manner as the associated samples, following the procedure described in Section 10.2.

12.1.2.3.2 Under no circumstances should method blanks be analyzed at a dilution.

12.1.2.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.0.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.2.2.

12.1.2.5.2 The %R of each of the DMCs in the blank must be within the acceptance windows in Table 10 – Deuterated Monitoring Compound Recovery Limits.

12.1.2.5.3 The blank must meet the sample acceptance criteria listed in Sections 11.3.4 - 11.3.7.

12.1.2.5.4 The concentration of each target analyte found in the method blank must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits, except for methylene chloride, acetone, and 2-butanone, which must be less than 2 times the respective CRQL.

12.1.2.5.5 The concentration of each TIC found in the method blank must be less than 0.5 µg/L.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.2.6.2 If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. It is the Contractor’s responsibility to ensure that method interferences caused by the contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in GCs be eliminated.

12.1.2.6.3 Any method blank that fails to meet the technical acceptance criteria must be reanalyzed. Further, all samples processed within the 12-hour period with a method blank that does not meet the blank technical acceptance criteria will require reanalysis at no additional cost to the EPA.

12.1.3 Instrument Blank

12.1.3.1 Summary of Instrument Blank

An instrument blank is a 25 mL aliquot of reagent water spiked with sufficient amount of internal standard spiking solution (Section 7.2.2.6) and DMC solution (Section 7.2.2.4.1), and carried through the entire analytical procedure. Instrument

blanks are analyzed after a sample/dilution that contains a target analyte exceeding the calibration range. The results from the instrument blank analysis indicate whether there is contamination from a previous sample.

12.1.3.2 Frequency of Instrument Blank

Samples may contain target analytes at levels exceeding the calibration. An instrument blank must be analyzed after the sample that exceeds the calibration range (also in the same purge inlet if an autosampler is used) or a sample that exceeds the maximum contamination criteria in Section 11.3.8 must be analyzed. If the instrument blank or sample does not meet the criteria (i.e., contaminated), the system must be decontaminated until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum carryover criteria.

NOTE: Only the instrument blank that demonstrates that there was no carryover from the previous sample or the instrument blank that demonstrates that the system is clean (Section 12.1.3.5.3) must be reported. Instrument blanks analyzed during the instrument decontamination process that exceed the requirements listed in Section 11.3.8 do not need to be reported.

12.1.3.3 Procedure for Instrument Blank

12.1.3.3.1 Instrument blanks shall be analyzed in the same manner as the associated samples following the procedures outlined in Section 10.0 and in accordance with the protocol of Section 11.3.8.

12.1.3.3.2 Under no circumstances should instrument blanks be analyzed at a dilution.

12.1.3.4 Calculations for Instrument Blank

Perform data analysis and calculations according to Section 11.0.

12.1.3.5 Technical Acceptance Criteria for Instrument Blank

12.1.3.5.1 All instrument blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.3.2.

12.1.3.5.2 The RT shift for each of the internal standards in the blank must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.1.3.5.3 The concentration of each target analyte in the instrument blank must be less than its CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits. The concentration of non-target compounds in blanks must be less than 2.0 ug/L.

12.1.3.5.4 It is the Contractor’s responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms, be eliminated.

12.1.3.6 Corrective Action for Instrument Blank

12.1.3.6.1 If a Contractor's instrument blanks exceed the criteria in Section 12.1.3.5, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds.

12.1.3.6.2 Any instrument blank that fails to meet the technical acceptance criteria described in Section 12.1.3.5 requires reanalysis of the samples analyzed after the instrument blank having any target analytes detected at levels above the CRQLs at no additional cost to the EPA.

12.1.4 Storage Blank

12.1.4.1 Summary of Storage Blank

A storage blank is a volume of reagent water. The vials are stored with the samples in the SDG under the same conditions. A 25 mL aliquot of this reagent water is spiked with internal standard spiking solution and DMC solution, and analyzed after all samples in the SDG have been analyzed. The storage blank indicates whether contamination may have occurred during storage of samples.

12.1.4.2 Frequency of Storage Blank

A minimum of one storage blank must be analyzed per SDG, after all samples for the SDG have been analyzed, unless the SDG contains only ampulated PE samples. Analysis of a storage blank is not required for SDGs that contain only ampulated PE samples.

12.1.4.3 Procedure for Storage Blank

12.1.4.3.1 Upon receipt of the first samples in an SDG, two 40 mL screw-cap VOA vials with a PTFE-faced silicone septum and are filled with reagent water are stored with the samples in the SDG under the same conditions.

12.1.4.3.2 Storage blank shall be analyzed in the same manner as the associated samples, following the procedure described in Section 10.0.

12.1.4.3.3 Under no circumstances should storage blanks be analyzed at a dilution.

12.1.4.4 Calculations for Storage Blank

Perform data analysis and calculations according to Section 11.0.

12.1.4.5 Technical Acceptance Criteria for Storage Blank

12.1.4.5.1 All storage blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.4.2.

12.1.4.5.2 The storage blank must be analyzed on a GC/MS system that also meets the technical acceptance criteria for the method blank.

12.1.4.5.3 The %R of each of the DMCs in the blank must be within the acceptance windows in Table 10 – Deuterated Monitoring Compound Recovery Limits.

12.1.4.5.4 The EICP area for each of the internal standards in the blank must be within the range of 50%-200% of its response in the most recent opening CCV standard analysis.

12.1.4.5.5 The RT shift for each of the internal standards in the blank must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.1.4.5.6 The concentration of each target analyte found in the storage blank must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits, except for methylene chloride, acetone, and 2-butanone, which must be less than 2 times the respective CRQL.

12.1.4.5.7 It is the Contractor's responsibility to ensure that method interferences caused by the contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.4.6 Corrective Action for Storage Blank

12.1.4.6.1 If a Contractor's storage blanks exceed the criteria in Section 12.1.4.5, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further analysis proceeds.

12.1.4.6.2 If the storage blank does not meet the technical acceptance criteria for blank analyses in Section 12.1.4.5, correct system problems and reanalyze the storage blank.

12.1.4.6.3 If, upon reanalysis, the storage blank meets the criteria, the problem occurred during the analysis and the reanalyzed storage blank results must be reported. If upon reanalysis, the storage blank still does not meet the criteria, the problem occurred during storage. The Laboratory Manager or their designee must address the problem in the SDG Narrative and discuss the corrective actions implemented to prevent future occurrences.

NOTE: A copy of the storage blank data must also be retained by the Contractor and be made available for inspection during on-site laboratory evaluations.

12.2 Matrix Spike and Matrix Spike Duplicate

12.2.1 Summary of Matrix Spike and Matrix Spike Duplicate

To evaluate the effects of the sample matrix on the method used for trace volatile analysis, the EPA has prescribed a mixture of volatile target analytes to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method. An MS/MSD shall only be analyzed if requested by the EPA Region (through SMO) or specified on the Traffic Report/Chain of Custody (TR/COC) Record.

12.2.2 Frequency of Matrix Spike and Matrix Spike Duplicate

12.2.2.1 If requested, an MS/MSD must be performed for each group of 20 field samples in an SDG, or each SDG, whichever is most frequent.

12.2.2.2 The Contractor shall not perform MS/MSD analysis on any of the field QC or PE samples.

12.2.2.3 If an insufficient number of sample vials were received to perform an MS/MSD, and MS/MSD are required, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the EPA Region for instructions. The EPA Region has the option to cancel the MS/MSD analysis. SMO will notify the Contractor of the resolution. The Contractor shall document the decision in the SDG Narrative.

12.2.2.4 If it appears that the EPA Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2.1, the Contractor shall contact SMO. SMO will contact the EPA Region to determine which samples should have an MS/MSD performed on them. SMO will notify the Contractor of the EPA Region's decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 When a Contractor receives only PE sample(s), no MS/MSD shall be performed within that SDG.

12.2.3 Procedure for Preparing Matrix Spike and Matrix Spike Duplicate

12.2.3.1 Add 10 µL of the matrix spiking solution (Section 7.2.2.5) to each of the 25 mL aliquots of the sample chosen for spiking. Process the samples according to Section 10. Disregarding any dilutions, this is equivalent to a concentration of 5 µg/L of each Matrix Spike analyte.

12.2.3.2 MS/MSD samples must be analyzed at the same dilution as the least diluted aliquot for which the original sample results will be reported to the EPA. Sample dilutions must be performed in accordance with Section 10.2.12. Do not further dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range.

12.2.4 Calculations for Matrix Spike and Matrix Spike Duplicate

12.2.4.1 Calculate the concentrations of the Matrix Spike analytes using the same equation as used for target analytes (Equation 7). Calculate the recovery of each Matrix Spike analyte using the following equation:

EQ. 10 Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each analyte in the MS/MSD using the following equation:

EQ. 11 Relative Percent Difference

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

WHERE,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

NOTE: The vertical bars in the equation above indicate the absolute value of the difference.

12.2.5 Technical Acceptance Criteria for Matrix Spike and Matrix Spike Duplicate

12.2.5.1 All MS/MSDs must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, CCV, and blank technical acceptance criteria, and at the frequency described in Section 12.2.2.

12.2.5.2 The MS/MSD must be analyzed within the contract holding time.

12.2.5.3 The RT shift for each of the internal standards in the MS/MSD must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.2.5.4 The limits for MS analyte recovery and RPD are given in Table 11 - Matrix Spike Recovery and Relative Percent Difference Limits. As these limits are only advisory, no further action by the Contractor is required.

12.2.6 Corrective Action for Matrix Spike and Matrix Spike Duplicate

Any MS/MSD that does not meet the technical acceptance criteria in Sections 12.2.5.1 and 12.2.5.3 must be reanalyzed at no additional cost to the EPA.

12.3 Laboratory Control Sample

Not applicable to this method.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed, the Method Detection Limit (MDL) for each trace volatile target analyte shall be determined on each instrument used for analysis. The MDLs must be determined annually thereafter or after major instrument maintenance. Major instrument maintenance includes, but is not limited to: cleaning or replacement of the mass spectrometer source, mass filters (e.g., quadrupole, ion trap, etc.), or electron multiplier (or similar device); and replacement or overhaul of the P/T device. A new MDL study will not be required after changing the GC column, as long as the replacement has the same length, inner diameter, and stationary phase.

12.4.2 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.3 The determined concentration of the MDL must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and submitted to the EPA within seven (7) days of study completion. This schedule and the designated recipients are specified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 of Exhibit D – Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 of Exhibit D – Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Method 524.2, Revision 4, August 1992.

16.2 U.S. Environmental Protection Agency, Purge-and-Trap for Aqueous Samples, Method 5030C, Revision 3, May 2003.

16.3 U.S. Environmental Protection Agency, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C, Revision 3, August 2006.

16.4 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS

Systematic Name	EPA Registry Name	Synonym	CAS #
Methane, dichlorodifluoro-	CFC-12	Dichlorodifluoromethane	75-71-8
Methane, chloro-	Chloromethane	Methyl chloride	74-87-3
Ethene, chloro-	Vinyl Chloride	Vinyl chloride	75-01-4
Methane, bromo-	Methyl Bromide	Methyl bromide	74-83-9
Ethane, chloro-	Chloroethane	Ethyl chloride	75-00-3
Methane, trichlorofluoro-	CFC-11	Fluorotrichloromethane	75-69-4
Ethene, 1,1-dichloro-	1,1-Dichloroethylene	Vinylidene chloride	75-35-4
Ethane, 1,1,2-trichloro-1,2,2-trifluoro-	CFC-113	Freon 113	76-13-1
2-Propanone	Acetone	Dimethyl ketone	67-64-1
Carbon disulfide	Carbon disulfide	Dithiocarbonic anhydride	75-15-0
Acetic acid, methyl ester	Methyl acetate	Methyl acetate	79-20-9
Methane, dichloro	Methylene chloride	Dichloromethane	75-09-2
Ethene, 1,2-dichloro-, (1E)-	trans-1,2-Dichloroethylene	Ethylene, 1,2-dichloro-, (E)-	156-60-5
Propane, 2-methoxy-2-methyl-	Methyl tert-butyl ether	t-Butyl methyl ether	1634-04-4
Ethane, 1,1-dichloro-	1,1-Dichloroethane	Ethylidene dichloride	75-34-3
Ethene, 1,2-dichloro-, (1Z)-	cis-1,2-Dichloroethylene	Ethylene, 1,2-dichloro-, (Z)-	156-59-2
2-Butanone	Methyl ethyl ketone	Butan-2-one	78-93-3
Methane, bromochloro-	Halon 1011	Chlorobromomethane	74-97-5
Methane, trichloro-	Chloroform	Trichloromethane	67-66-3
Ethane, 1,1,1-trichloro-	1,1,1-Trichloroethane	1,1,1-TCE	71-55-6
Cyclohexane	Cyclohexane	Hexahydrobenzene	110-82-7
Methane, tetrachloro-	Carbon tetrachloride	Tetrachlorocarbon	56-23-5
Benzene	Benzene	Benzol	71-43-2
Ethane, 1,2-dichloro-	1,2-Dichloroethane	Ethylene dichloride	107-06-2
Ethene, 1,1,2-trichloro-	Trichloroethylene	Ethylene, trichloro-	79-01-6
Cyclohexane, methyl-	Methylcyclohexane	Hexahydrotoluene	108-87-2
Propane, 1,2-dichloro-	1,2-Dichloropropane	Propylene dichloride	78-87-5

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
Methane, bromodichloro-	Dichlorobromomethane	Bromodichloromethane	75-27-4
1-Propene, 1,3-dichloro-, (Z)-	cis-1,3-Dichloropropene	cis-1,3-Dichloropropylene	10061-01-5
2-Pentanone, 4-methyl-	Methyl isobutyl ketone	2-Methylpropyl methyl ketone	108-10-1
Benzene, methyl-	Toluene	Methylbenzol	108-88-3
1-Propene, 1,3-dichloro-, (1E)-	trans-1,3-Dichloropropene	trans-1,3-Dichloropropylene	10061-02-6
Ethane, 1,1,2-trichloro-	1,1,2-Trichloroethane	1,1,2-TCA	79-00-5
Ethene, 1,1,2,2-tetrachloro-	Tetrachloroethylene	Tetrachlorethene	127-18-4
2-Hexanone	2-Hexanone	Methyl n-butyl ketone	591-78-6
Methane, dibromochloro-	Chlorodibromomethane	Dibromochloromethane	124-48-1
Ethane, 1,2-dibromo-	Ethylene Dibromide	1,2-Dibromoethane	106-93-4
Benzene, chloro-	Chlorobenzene	Phenyl chloride	108-90-7
Benzene, ethyl-	Ethylbenzene	Phenylethane	100-41-4
Benzene, 1,2-dimethyl-	o-Xylene	1,2-Dimethylbenzene	95-47-6
Benzene, (1,3 and 1,4)-dimethyl-	m,p-Xylene	(1,3 and 1,4)-Dimethyl benzene	179601-23-1
Benzene, ethenyl-	Styrene	Vinyl Benzene	100-42-5
Methane, tribromo-	Tribromomethane	Bromoform	75-25-2
Benzene, (1-methylethyl)-	Cumene	Isopropylbenzene	98-82-8
Ethane, 1,1,2,2-tetrachloro-	1,1,2,2-Tetrachloroethane	Acetylene tetrachloride	79-34-5
Benzene, 1,3-dichloro-	m-Dichlorobenzene	m-Phenylene dichloride	541-73-1
Benzene, 1,4-dichloro-	p-Dichlorobenzene	p-Chlorophenyl chloride	106-46-7
Benzene, 1,2-dichloro-	o-Dichlorobenzene	ortho-Dichlorobenzene	95-50-1
Propane, 1,2-dibromo-3-chloro-	1,2-Dibromo-3-chloropropane	Dibromochloropropane	96-12-8
Benzene, 1,2,4-trichloro-	1,2,4-Trichlorobenzene	1,2,4-Trichlorobenzol	120-82-1
Benzene, 1,2,3-trichloro-	1,2,3-Trichlorobenzene	Vic-Trichlorobenzene	87-61-6
Internal Standards			
Benzene-d5, chloro-	Chlorobenzene-d5	Chlorobenzene-d5	3114-55-4
Benzene, 1,4-difluoro	1,4-Difluorobenzene	p-Difluorobenzene	540-36-3
Benzene-1,2,4,5-d4, 3,6-dichloro	1,4-Dichlorobenzene-d4	1,4-Dichloro-2,3,5,6-tetradeuterobenzene	3855-82-1

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
DMCs			
Ethene-d3, chloro-	Vinyl chloride-d3	Vinyl chloride-d3	6745-35-3
Ethane-d5, chloro-	Chloroethane-d5	Chloroethane-d5	19199-91-8
Ethene-1,1-d2, dichloro-	1,1-Dichloroethene-d2	1,1-Dichloroethene-d2	22280-73-5
2-Butanone-1,1,1,3,3-d5	2-Butanone-d5	2-Butanone-d5	24313-50-6
Methane-d, trichloro-	Chloroform-d	Chloroform-d	865-49-6
Ethane-1,1,2,2-d4, 1,2-dichloro-	1,2-Dichloroethane-d4	1,2-Dichloroethane-d4	17060-07-0
Benzene-1,2,3,4,5,6-d6	Benzene-d6	Benzene-d6	1076-43-3
Propane-1,1,1,2,3,3-d6, 2,3-dichloro-	1,2-Dichloropropane-d6	1,2-Dichloropropane-d6	93952-08-0
Benzene-d5, methyl-d3-	Toluene-d8	Perdeuterotoluene	2037-26-5
1-Propene-1,2,3,3-d4, 1,3-dichloro-(E)-	Trans-1,3-Dichloropropene-d4	Trans-1,3-Dichloropropene-d4	93951-86-1
2-Hexanone-1,1,1,3,3-d5		2-Hexanone-d5	4840-82-8
Ethane-1,2-d2, 1,1,2,2-tetrachloro-	1,1,2,2-Tetrachloroethane-d2	1,1,2,2-Tetrachloroethane-d2	33685-54-0
Benzene-1,2,3,4-d4, 5,6-dichloro-	1,2-Dichlorobenzene-d4	1,2-Dichloro-3,4,5,6-tetradeuterobenzene	2199-69-1

TABLE 2. 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 80.0% of mass 95
95	base peak, 100% Relative Abundance
96	5.0 - 9.0% of mass 95 (see NOTE)
173	less than 2.0% of mass 174
174	50.0 - 120% of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101% of mass 174
177	5.0 - 9.0% of mass 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

TABLE 3. TRACE VOLATILE DEUTERATED MONITORING COMPOUNDS
AND THE ASSOCIATED TARGET ANALYTES

Vinyl chloride-d_3 (DMC-1)	**Chloroethane-d_5 (DMC-2)**	**1,1-Dichloroethene-d_2 (DMC-3)**
Vinyl chloride	Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon disulfide	trans-1,2-Dichloroethene cis-1,2-Dichloroethene 1,1-Dichloroethene
2-Butanone-d_5 (DMC-4)	**Chloroform-d (DMC-5)**	**1,2-Dichloroethane-d_4 (DMC-6)**
Acetone 2-Butanone	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
Benzene-d_6 (DMC-7)	**1,2-Dichloropropane-d_6 (DMC-8)**	**Toluene-d_8 (DMC-9)**
Benzene	Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene-d_4 (DMC-10)	**2-Hexanone-d_5 (DMC-11)**	**1,1,2,2-Tetrachloroethane-d_2 (DMC-12)**
cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	4-Methyl-2-pentanone 2-Hexanone	1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene-d_4 (DMC-13)		
Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene		

TABLE 4. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR TRACE VOLATILE ORGANIC COMPOUNDS

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
Dichlorodifluoromethane	0.010	0.010	30.0	±40.0	±50.0
Chloromethane	0.010	0.010	30.0	±30.0	±50.0
Vinyl chloride	0.010	0.010	30.0	±30.0	±50.0
Bromomethane	0.010	0.010	40.0	±30.0	±50.0
Chloroethane	0.010	0.010	30.0	±30.0	±50.0
Trichlorofluoromethane	0.010	0.010	30.0	±30.0	±50.0
1,1-Dichloroethene	0.020	0.020	30.0	±20.0	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	0.010	30.0	±30.0	±50.0
Acetone	0.010	0.010	40.0	±40.0	±50.0
Carbon disulfide	0.010	0.010	20.0	±25.0	±25.0
Methyl acetate	0.010	0.010	40.0	±40.0	±50.0
Methylene chloride	0.010	0.010	40.0	±30.0	±50.0
trans-1,2-Dichloroethene	0.070	0.070	20.0	±20.0	±25.0
Methyl tert-butyl ether	0.010	0.010	30.0	±30.0	±50.0
1,1-Dichloroethane	0.100	0.100	20.0	±20.0	±25.0
cis-1,2-Dichloroethene	0.100	0.100	20.0	±20.0	±25.0
2-Butanone	0.010	0.010	40.0	±40.0	±50.0
Bromochloromethane	0.020	0.020	20.0	±20.0	±25.0
Chloroform	0.040	0.040	20.0	±20.0	±25.0
1,1,1-Trichloroethane	0.050	0.050	30.0	±20.0	±25.0
Cyclohexane	0.100	0.100	30.0	±25.0	±50.0
Carbon tetrachloride	0.020	0.020	20.0	±25.0	±50.0
Benzene	0.300	0.300	20.0	±20.0	±25.0
1,2-Dichloroethane	0.010	0.010	20.0	±25.0	±50.0
Trichloroethene	0.100	0.100	20.0	±20.0	±25.0
Methylcyclohexane	0.200	0.200	30.0	±25.0	±50.0
1,2-Dichloropropane	0.100	0.100	20.0	±20.0	±25.0
Bromodichloromethane	0.090	0.090	20.0	±20.0	±25.0
cis-1,3-Dichloropropene	0.100	0.100	20.0	±20.0	±25.0
4-Methyl-2-pentanone	0.010	0.010	30.0	±30.0	±50.0
Toluene	0.400	0.400	20.0	±20.0	±25.0
trans-1,3-Dichloropropene	0.010	0.010	30.0	±20.0	±25.0
1,1,2-Trichloroethane	0.040	0.040	20.0	±20.0	±25.0
Tetrachloroethene	0.100	0.100	20.0	±20.0	±25.0
2-Hexanone	0.010	0.010	40.0	±40.0	±50.0
Dibromochloromethane	0.050	0.050	20.0	±20.0	±25.0

TABLE 4. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR TRACE VOLATILE ORGANIC COMPOUNDS (CON'T)

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
1,2-Dibromoethane	0.010	0.010	20.0	±20.0	±25.0
Chlorobenzene	0.400	0.400	20.0	±20.0	±25.0
Ethylbenzene	0.500	0.500	20.0	±20.0	±25.0
m,p-Xylene	0.200	0.200	20.0	±20.0	±25.0
o-Xylene	0.300	0.300	30.0	±20.0	±25.0
Styrene	0.200	0.200	30.0	±20.0	±25.0
Bromoform	0.010	0.010	30.0	±30.0	±50.0
Isopropylbenzene	0.700	0.700	30.0	±25.0	±25.0
1,1,2,2-Tetrachloroethane	0.050	0.050	20.0	±25.0	±25.0
1,3-Dichlorobenzene	0.500	0.500	20.0	±20.0	±25.0
1,4-Dichlorobenzene	0.700	0.700	20.0	±20.0	±25.0
1,2-Dichlorobenzene	0.400	0.400	20.0	±20.0	±25.0
1,2-Dibromo-3-chloropropane	0.010	0.010	40.0	±40.0	±50.0
1,2,4-Trichlorobenzene	0.300	0.300	30.0	±30.0	±50.0
1,2,3-Trichlorobenzene	0.200	0.200	30.0	±40.0	±50.0
Deuterated Monitoring Compounds					
Vinyl chloride-d_3	0.010	0.010	30.0	±30.0	±50.0
Chloroethane-d_5	0.010	0.010	30.0	±30.0	±50.0
1,1-Dichloroethene-d_2	0.010	0.010	30.0	±25.0	±25.0
2-Butanone-d_5	0.010	0.010	40.0	±40.0	±50.0
Chloroform-d	0.010	0.010	20.0	±20.0	±25.0
1,2-Dichloroethane-d_4	0.010	0.010	20.0	±25.0	±25.0
Benzene-d_6	0.030	0.030	20.0	±20.0	±25.0
1,2-Dichloropropane-d_6	0.100	0.100	20.0	±20.0	±25.0
Toluene-d_8	0.200	0.200	20.0	±20.0	±25.0
trans-1,3-Dichloropropene-d_4	0.010	0.010	30.0	±25.0	±25.0
2-Hexanone-d_5	0.010	0.010	40.0	±40.0	±50.0
1,1,2,2-Tetrachloroethane-d_2	0.010	0.010	20.0	±25.0	±25.0
1,2-Dichlorobenzene-d_4	0.060	0.060	20.0	±20.0	±25.0

[1] If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

TABLE 5. PURGE-AND-TRAP ANALYTICAL CONDITIONS

Purge Conditions	
Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ±0.1 min.
Purge Flow Rate:	25-40 mL/min.
Purge Temperature:	Ambient temperature
Desorb Conditions	
Desorb Temperature:	180°C
Desorb Flow Rate:	15 mL/min.
Desorb Time:	4.0 ±0.1 min.
Trap Reconditioning Conditions	
Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 ±0.1 min. (minimum). A longer time may be required to bake contamination or water from the system.

NOTE: Higher purge temperatures may be used provided that manufacturer's instructions are followed and technical acceptance criteria are met for all standards, samples, and blanks. Certain target analytes, such as methyl tert-butyl ether (MTBE), may decompose at high purge temperatures in samples that have been acid preserved.

TABLE 6. GAS CHROMATOGRAPH ANALYTICAL CONDITIONS

Capillary Columns	
Carrier Gas:	Helium
Flow Rate:	15 mL/min.
Initial Temperature:	10°C
Initial Hold Time:	1.0 – 5.0 (±0.1) min.
Ramp Rate:	6°C/min.
Final Temperature:	160°C
Final Hold Time:	Until 3 min. after all analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 1 – Trace Volatiles Target Analyte List and Contract Required Quantitation Limits, elute (required)

TABLE 7. MASS SPECTROMETER ANALYTICAL CONDITIONS

Electron Energy	70 volts (nominal)
Mass Range	35-300 u
Ionization Mode	Electron ionization (EI)
Scan Time	To give at least 5 scans per peak, not to exceed 2 sec. per scan.

TABLE 8. CHARACTERISTIC IONS FOR TRACE VOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,63
1,1,2-Trichloro-1,2,2-trifluoroethane	101	85,151
Acetone	43	58
Carbon disulfide	76	78
Methyl acetate	43	74
Methylene chloride	84	49,86
trans-1,2-Dichloroethene	96	61,98
Methyl tert-butyl ether	73	43,57
1,1-Dichloroethane	63	65,83
cis-1,2-Dichloroethene	96	61,98
2-Butanone	43*	72
Chloroform	83	85
Bromochloromethane	128	49,130,51
1,1,1-Trichloroethane	97	99,61
Cyclohexane	56	69,84
Carbon tetrachloride	117	119
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	95	97,132,130
Methylcyclohexane	83	55,98
1,2-Dichloropropane	63	112
Bromodichloromethane	83	85,127
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58,100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	83,85,99,132,134
Tetrachloroethene	164	129,131,166
2-Hexanone	43	58,57,100
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Chlorobenzene	112	77,114
Ethylbenzene	91	106
m,p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78

*m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TABLE 8. CHARACTERISTIC IONS FOR TRACE VOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS (CON'T)

Analyte	**Primary Quantitation Ion**	**Secondary Ion(s)**
Bromoform	173	175,254
Isopropylbenzene	105	120,77
1,1,2,2-Tetrachloroethane	83	85,131
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene	146	111,148
1,2-Dibromo-3-chloropropane	75	157,155
1,2,4-Trichlorobenzene	180	182,145
1,2,3-Trichlorobenzene	180	182,145
Deuterated Monitoring Compounds		
Vinyl chloride-d_3	65	67
Chloroethane-d_5	69	71,51
1,1-Dichloroethene-d_2	63	98,65
2-Butanone-d_5	46	77
Chloroform-d	84	86,47,49
1,2-Dichloroethane-d_4	65	67,51
Benzene-d_6	84	82,54,52
1,2-Dichloropropane-d_6	67	65,46,42
Toluene-d_8	98	100,42
trans-1,3-Dichloropropene-d_4	79	81,42
2-Hexanone-d_5	63	46
1,1,2,2-Tetrachloroethane-d_2	84	86
1,2-Dichlorobenzene-d_4	152	150
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115,150
1,4-Difluorobenzene	114	63,88
Chlorobenzene-d_5	117	82,119

TABLE 9. TRACE VOLATILE TARGET ANALYTES AND DEUTERATED MONITORING COMPOUNDS WITH ASSOCIATED INTERNAL STANDARDS FOR QUANTITATION

1,4-Difluorobenzene (IS)	**Chlorobenzene-d_5 (IS)**	**1,4-Dichlorobenzene-d_4 (IS)**
Dichlorodifluoromethane	1,1,1-Trichloroethane	Bromoform
Chloromethane	Cyclohexane	1,3-Dichlorobenzene
Vinyl chloride	Carbon tetrachloride	1,4-Dichlorobenzene
Bromomethane	Benzene	1,2-Dichlorobenzene
Chloroethane	Trichloroethene	1,2-Dibromo-3-chloropropane
Trichlorofluoromethane	Methylcyclohexane	
1,1-Dichloroethene	1,2-Dichloropropane	1,2,4-Trichlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Bromodichloromethane	1,2,3-Trichlorobenzene
	cis-1,3-Dichloropropene	1,2-Dichlorobenzene-d_4 (DMC)
Acetone	4-Methyl-2-pentanone	
Carbon disulfide	Toluene	
Methyl acetate	trans-1,3-Dichloropropene	
Bromochloromethane	1,1,2-Trichloroethane	
Methylene chloride	Tetrachloroethene	
trans-1,2-Dichloroethene	2-Hexanone	
Methyl tert-butyl ether	Dibromochloromethane	
1,1-Dichloroethane	1,2-Dibromoethane	
cis-1,2-Dichloroethene	Chlorobenzene	
2-Butanone	Ethylbenzene	
Chloroform	m,p-Xylene	
1,2-Dichloroethane	o-Xylene	
Vinyl chloride-d_3 (DMC)	Styrene	
Chloroethane-d_5 (DMC)	Isopropylbenzene	
1,1-Dichloroethene-d_2 (DMC)	1,1,2,2-Tetrachloroethane	
	Benzene-d_6 (DMC)	
2-Butanone-d_5 (DMC)	1,2-Dichloropropane-d_6 (DMC)	
Chloroform-d (DMC)	trans-1,3-Dichloropropene-d_4 (DMC)	
1,2-Dichloroethane-d_4 (DMC)	Toluene-d_8 (DMC)	
	2-Hexanone-d_5 (DMC)	
	1,1,2,2-Tetrachloroethane-d_2 (DMC)	

TABLE 10. DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

Compound	Percent Recovery Limits
Vinyl chloride-d_3	40-130
Chloroethane-d_5	65-130
1,1-Dichloroethene-d_2	60-125
2-Butanone-d_5	40-130
Chloroform-d	70-125
1,2-Dichloroethane-d_4	70-130
Benzene-d_6	70-125
1,2-Dichloropropane-d_6	60-140
Toluene-d_8	70-130
trans-1,3-Dichloropropene-d_4	55-130
2-Hexanone-d_5	45-130
1,1,2,2-Tetrachloroethane-d_2	65-120
1,2-Dichlorobenzene-d_4	80-120

NOTE: The recovery limits for any of the compounds listed above may be expanded at any time during the period of performance if the EPA determines that the limits are too restrictive.

TABLE 11. MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Percent Recovery	RPD
1,1-Dichloroethene	61-145	0-14
Benzene	76-127	0-11
Trichloroethene	71-120	0-14
Toluene	76-125	0-13
Chlorobenzene	75-130	0-13

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EXHIBIT D

LOW/MEDIUM CONCENTRATIONS OF
VOLATILE ORGANIC COMPOUNDS ANALYSIS

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Exhibit D – Low/Medium Concentrations of
Volatile Organic Compounds Analysis

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Exhibit D – Low/Medium Concentrations of
Volatile Organic Compounds Analysis

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water, leachate derived from the Toxicity Characteristics Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP), and soil/sediment samples from hazardous waste sites for the volatile organic compounds in the Target Analyte List (TAL) for low/medium volatiles in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits. The method, based on U.S. Environmental Protection Agency (EPA) Method 8260C, includes sample preparation and analysis to determine the approximate concentration of volatile organic constituents in the sample. The actual analysis is based on a purge-and-trap (P/T) Gas Chromatograph/Mass Spectrometer (GC/MS) method for aqueous and medium-level soil samples and closed-system purge-and-trap for low-level soil samples.

1.2 Problems that have been associated with the following analytes using this method include:

- Chloromethane, vinyl chloride, bromomethane, and chloroethane may display peak broadening if the analytes are not delivered to the GC column in a tight band.
- Acetone, hexanone, 2-butanone, and 4-methyl-2-pentanone have poor purge efficiencies and may be lost if purge flow is too slow.
- 1,1,1-trichloroethene and all of the dichloroethenes may dehydrohalogenate during storage or analysis.
- Tetrachloroethane and 1,1-dichloroethane may be degraded by contaminated transfer lines in P/T systems and/or active sites in trapping materials.
- Chloromethane and other gases may be lost if the purge flow is too fast.
- Bromoform is one of the analytes most likely to be adversely affected by cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by tuning of 4-bromofluorobenzene (BFB) at ions m/z 174/176. Increasing the m/z 174/176 ratio within the specified Quality Control (QC) limits may improve bromoform response.

2.0 SUMMARY OF METHOD

2.1 Water/TCLP or SPLP Leachate

An inert gas is bubbled through a 5 milliliter (mL) sample contained in a specially designed purging chamber at ambient temperature. Higher purge temperatures may be used, provided that all technical acceptance criteria are met for all standards, samples, and blanks. The same purge conditions must be used for all associated standards, samples, and blanks. The purgeable compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeable compounds are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeable compounds onto a GC wide-bore capillary column. The GC is temperature-programmed to separate the purgeable compounds, which are then detected with an MS.

2.2 Soil/Sediment

2.2.1 Low-Level Soil/Sediment

Low-level volatile organic compounds are generally determined by analyzing approximately 5 grams (g) of sample in a pre-weighed vial with a septum-sealed screw-cap (Section 6.1.10) that already contains a stirring bar.

NOTE: The sodium bisulfate preservative may be used under limited circumstances. 5 mL of sodium bisulfate solution (Section 7.1.3) is added to each sample when preservation by sodium bisulfate is requested by the EPA Region.

The entire vial is placed into the instrument carousel. Immediately before analysis, organic-free reagent water, Deuterated Monitoring Compounds (DMCs), and internal standards are automatically added without opening the sample vial. The vial containing the sample is heated to the suggested temperature of 40°C and the volatiles are purged through a sorbent trap using an inert gas combined with agitation of the sample. Higher purge temperatures may be required for the analysis of certain target analytes. When purging is complete, the sorbent column is heated and backflushed with helium to desorb the purgeable compounds onto a capillary GC column. The GC is temperature-programmed to separate the purgeable compounds, which are then detected with an MS.

2.2.2 Medium-Level Soil/Sediment

A soil sample of 5 g is collected, preserved in methanol, and/or extracted with methanol. An aliquot of the methanol extract is added to 5 mL of reagent water. An inert gas is bubbled through this solution in a specially designed purging chamber at ambient temperature. The purgeable compounds are effectively transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeable compounds onto a capillary GC column. The GC is temperature-programmed to separate the purgeable compounds, which are then detected with an MS.

2.3 Wipes

Not applicable to this method.

2.4 Waste

Not applicable to this method.

2.5 Non-Target Compounds

Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library. Non-target compounds are quantitated by comparing the area response from the total Reconstructed Ion Chromatogram (RIC) for the non-target compound peaks to the area response provided by the nearest internal standard compound. A Relative Response Factor (RRF) of 1 is assumed.

3.0 DEFINITIONS

See Exhibit G – Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

4.1.1 Method interference may be caused by impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by analyzing laboratory method and instrument blanks as described in Section 12.1. The use of non-polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.1.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples must be stored separately from other laboratory samples and standards, and must be analyzed in a room whose atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis.

4.1.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank, or the next sample must be closely monitored to check for cross-contamination. 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 a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

4.1.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine the presence of methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all Gas Chromatography (GC) carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken. At the time of sample receipt, the Contractor must prepare two 40 mL VOA vials containing reagent water and/or inert sand to be stored as storage blanks with each group of samples (Section 12.1.4).

4.2 Matrix Interferences

Matrix interferences may be caused by compounds that are purged or co-extracted from the sample. The extent of matrix interferences will vary considerably depending on the nature of the site being sampled.

5.0 SAFETY

See Section 12.0 of Exhibit D – Introduction to Organic Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternative equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 General Laboratory Equipment

6.1.1 Balances

6.1.1.1 Top loading, capable of weighing accurately to ±0.01 g.

6.1.1.2 Analytical, capable of weighing accurately to ±0.0001 g.

6.1.1.3 A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each type of balance and be accurate to ±0.01 g and ±0.0001 g, respectively. The masses that are used to check the balances daily must be checked on a monthly basis using NIST-traceable known reference masses (Class '0' or Class '1') as defined by ASTM E617-97(2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified at least every five years, or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates these criteria have been met.

6.1.2 Bottle - 15 mL, screw-cap, with PTFE cap liner.

6.1.3 Magnetic Stirring Bars - PTFE or glass-coated, of the appropriate size to fit the sample vials. Consult the manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturer of the purging device and the stirring bars for suggested cleaning procedures.

6.1.4 Micro Syringes - 25 microliters (µL) with a 2 inch x 0.006 inch ID, 22 gauge beveled needle. 10 µL and 100 µL. All micro syringes shall be visually inspected and documented monthly.

6.1.5 Pasteur Pipettes, Disposable.

6.1.6 pH Paper - Wide range.

6.1.7 Spatula – Stainless steel or PTFE.

6.1.8 Syringes - 25 mL glass hypodermic syringes with a Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used). 5.0, 1.0, and 0.5 mL syringes, gas-tight with shut-off valve.

6.1.9 Syringe Valve - Two-way, with Luer-Lok ends (three each), if applicable to the purging device.

6.1.10 Vials

6.1.10.1 40 mL, screw-cap, PTFE-lined, septum-sealed glass vials. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

6.1.10.2 60 mL, septum-sealed glass vials to collect samples for screening, percent moisture determination.

6.1.10.3 Vials and Caps - Assorted sizes.

6.1.11 Volumetric Flasks - Class A, 10 mL and 100 mL, with ground-glass stoppers.

6.2 Glassware/Extraction/Cleanup Equipment

Not applicable to this method.

6.3 Analytical Instrumentation

6.3.1 Gas Chromatograph

The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout desorption and temperature program operations. The system must include or be interfaced to a P/T system as specified in Section 6.3.4 and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants, or flow controllers with rubber components, are not to be used.

6.3.2 Gas Chromatography Columns

Recommended Column: Minimum length 30 meter (m) x 0.53 mm ID fused silica wide-bore capillary column with a 6% Cyanopropylphenyl 94% Dimethyl Polysiloxane phase having a 3 micrometer (µm) film thickness (i.e., VOCOL, Rtx®-502.2, DB-624, Rtx®-624, CP-Select 624CB, or equivalent fused silica wide-bore capillary column). A description of the column used for analysis shall be provided in the SDG Narrative. Packed GC columns cannot be used.

The column shall accept up to 1000 nanograms (ng) of each analyte listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 - Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits without becoming overloaded.

6.3.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants that interfere with the identification and quantitation of the analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.
- The analytical results generated using the column meet the initial calibration, initial calibration verification (ICV), and continuing calibration verification (CCV) technical acceptance criteria (Sections 9.3.5, 9.4.5, and 9.5.5) and the Contract Required Quantitation Limits (CRQLs) listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits. Sufficient chromatographic resolution is achieved when the

height of the valley between two isomer peaks is less than 50% of the average of the two peak heights.

- The column provides equal or better resolution of the analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits than the columns listed in Section 6.3.2.

6.3.2.1.1 As applicable, follow the manufacturer's instructions for use of its product.

6.3.2.1.2 The Contractor must maintain documentation that the column met the criteria in Section 6.3.2.1. The minimum documentation is as follows:

6.3.2.1.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.3.2.1.2.2 RICs and data system reports generated on the GC/MS used for EPA Contract Laboratory Program (CLP) analyses:

- From instrument blanks that demonstrate that there are no contaminants that interfere with the volatile analysis when using the alternate column; and
- From initial calibration, ICV, and CCV standards analyzed using the alternate column.

6.3.2.1.3 Based on the Contractor-generated data described above, the Contractor shall complete a written comparison/review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Sections 9.3.5, 9.4.5, and 9.5.5;
- The low-point initial calibration standard analysis has adequate sensitivity to meet the volatile CRQLs;
- The high-point initial calibration standard analysis was not overloaded; and
- The column does not introduce contaminants that interfere with the identification and/or quantitation of analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.

6.3.2.1.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request by the EPA Regional CLP Contracting Officer's Representative (COR).

6.3.3 Mass Spectrometer

The MS must be capable of scanning from 35-300 atomic mass units (u) every 2 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the BFB GC/MS performance check technical acceptance criteria in Table 2 – 4-Bromofluorobenzene Key Ions and Ion Abundance Criteria, when 50 ng of BFB is injected through the GC inlet. The instrument conditions required for the acquisition of the BFB mass spectrum are given in Section 9.2.4.

NOTE: To ensure sufficient precision of mass spectral data, the MS scan rate should allow acquisition of at least five spectra while a sample compound elutes from the GC. The P/T GC/MS system must be in a room whose atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. The instrument must be vented to the outside of the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.3.3.1 Gas Chromatograph/Mass Spectrometer Interface

Any GC/MS interface may be used that gives acceptable calibration points at 25 ng or less per injection for each of the purgeable non-ketone target analytes and DMCs, and achieves all acceptable performance criteria. GC/MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.3.4 Purge-and-Trap Device

The P/T device consists of three separate pieces of equipment: the sample purge chamber, the trap, and the desorber. The analyst either manually or automatically (through an automated P/T device separate or integral with the GC) samples an appropriate volume (e.g., 5.0 mL) from the vial; adds DMCs, matrix spikes (MS), and internal standards to the sample; and transfers the sample to the purge device. The device also purges volatile organic compounds (VOCs) using an inert gas stream and traps the released VOCs for subsequent desorption into the GC. For low-level soil samples, the P/T device consists of a unit that automatically adds water, DMC spiking solution, and internal standard spiking solution to a hermetically-sealed vial containing the sample; purges the volatile target analytes using an inert gas stream while agitating the contents of the vial; and traps the released volatile target analytes for subsequent desorption into the GC. Such systems shall meet the following specifications:

6.3.4.1 The P/T device must be capable of accepting 40 mL closed-system P/T sample vials from the field, which are not to be opened during the analytical process.

6.3.4.2 The specific required sample containers will depend on the P/T system to be employed. The Contractor shall consult the P/T system manufacturer’s instructions regarding suitable specific vials, septa, caps, and mechanical agitation devices.

6.3.4.3 The sample purge chamber must be designed to accept 5.0 mL samples with a water column at least 3 centimeters (cm) deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

6.3.4.4 For soil samples, the purging device should be capable of accepting a vial large enough to contain a 5 g soil/sediment sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging (e.g., using a magnetic stirring bar, sonication, or

other means). The analytes being purged must be quantitatively transferred to an adsorber trap. The trap must be capable of transferring the adsorbed volatile compounds to the GC.

6.3.4.5 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inches (2.667 mm). The trap must be packed to contain (starting from the inlet) 0.5 cm silanized glass wool, and the following minimum lengths of adsorbent:

- 8 cm of 2,6-diphenylene oxide polymer (60/80 mesh chromatographic grade Tenax GC or equivalent).
- 1 cm methyl silicone packing, 3.0% OV-1 on Chromasorb W, 60/80 mesh (or equivalent).
- 8 cm of silica gel, 35/60 mesh (or equivalent).
- 7 cm of coconut charcoal.

6.3.4.6 Alternate sorbent traps may be used if:

- The trap packing materials do not introduce contaminants that interfere with identification and quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated using the trap meet the initial calibration, ICV, and CCV technical acceptance criteria listed in the analytical method and the CRQLs listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 - Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits; and
- The trap must be capable of accepting up to 1000 ng of each analyte listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 - Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits without becoming overloaded.

6.3.4.6.1 Before use of any trap other than the one specified in Section 6.3.4.5, the Contractor must first meet the criteria listed in Section 6.3.4.6. Once this has been demonstrated, the Contractor must document its use in each SDG Narrative by specifying the trap composition (packing material/brand name, amount of packing material). Other sorbent traps include, but are not limited to: Tenax/Silica Gel/Carbon Trap from EPA Method 524.2 and Vocarb 4000 Trap (Supelco) or equivalent.

6.3.4.6.2 The Contractor must maintain documentation that the alternate trap meets the criteria listed in Section 6.3.4.6. The minimum documentation requirements are as follows:

6.3.4.6.2.1 Manufacturer provided information concerning the performance characteristics of the trap.

6.3.4.6.2.2 RICs and data system reports generated on the Contractor’s GC/MS used for CLP analyses:

- From instrument blank analyses that demonstrate that there are no contaminants that interfere with the volatile analysis when using the alternate trap; and
- From initial calibration, ICV, and CCV standards analyzed using the trap specified in Section 6.3.4.

6.3.4.6.2.3 Based on Contractor-generated data described above, the Contractor must complete a written comparison/review that has been signed by the Laboratory Manager certifying that:

- The alternate trap performance meets the technical acceptance criteria listed in Sections 9.3.5, 9.4.5, and 9.5.5;
- The low-point initial calibration standard analysis has adequate sensitivity to meet the low/medium volatile CRQLs;
- The high-point initial calibration standard analysis was not overloaded; and
- The alternate trap materials do not introduce contaminants that interfere with the identification and/or quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.

6.3.4.6.2.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request of the EPA Regional CLP COR.

6.3.4.6.2.5 A description of the trap used for analysis shall be provided in the SDG Narrative.

6.3.4.7 The P/T apparatus may be assembled as a separate unit or be an integral unit coupled with a GC.

6.3.4.8 The desorber shall be capable of rapidly heating the trap to the desorb temperature recommended for the trap in use. The polymer section of the trap should not be heated higher than 180°C and the remaining sections should not exceed 220°C during bake-out mode.

6.4 Data Systems/Data Storage

A computer system must be interfaced to the MS that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows comparing sample spectra against reference library spectra. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

7.0 REAGENTS AND STANDARDS

The Contractor must provide all standards to be used with the contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Organic Analytical Methods, Section 11. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1 Reagents

7.1.1 Reagent Water - Reagent water is defined as water in which an interferent is not observed at or above the CRQL for each analyte of interest.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g [1 pound (lb)] of activated carbon.

7.1.1.2 Reagent water may also be generated using a water purification system.

7.1.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle, seal with a PTFE-lined septum, and cap.

7.1.2 Methanol - High Performance Liquid Chromatography (HPLC) quality or equivalent - Each lot of methanol used for analysis under the contract must be purged with nitrogen and must be demonstrated to be free of contaminants that interfere with the measurement of purgeable analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.

7.1.3 Sodium Bisulfate Solution - 2 g of ACS reagent grade or equivalent sodium bisulfate is dissolved for every 5 g of water.

7.2 Standards

7.2.1 Stock Standard Solutions

Stock standard solutions are defined as standards that are to be used to produce working standards. They may be in the form of single compounds or mixtures. They may be in methanol from pure standard materials or purchased as pre-made solutions. Prepare fresh stock standards every 6 months, or sooner if the standard has degraded or evaporated.

7.2.2 Working Standards

7.2.2.1 Initial and Continuing Calibration Solutions

Prepare working calibration standard solution(s) containing all of the purgeable target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits) in methanol. Prepare fresh calibration

standard solution(s) every month, or sooner if the solution has degraded or evaporated.

NOTE: The Contractor may prepare a calibration standard containing all of the non-ketones and a separate standard containing ketones.

7.2.2.1.1 Add a sufficient amount of each working standard to a 5.0 mL aliquot of reagent water to produce the desired calibration standard concentrations listed in Section 7.2.2.1.2 or 7.2.2.1.4.

7.2.2.1.2 Prepare five aqueous initial calibration standard solutions containing all of the purgeable target analytes and the DMCs at the following levels: all non-ketone target analytes and their associated DMCs (see Table 3 – Volatile Deuterated Monitoring Compounds and the Associated Target Analytes) at 5.0, 10, 50, 100, and 200 µg/L; all ketones and their associated DMCs (see Table 3 - Volatile Deuterated Monitoring Compounds and the Associated Target Analytes) at 10, 20, 100, 200, and 400 µg/L. All three xylene isomers (o-, m-, and p-xylene) must be present in the calibration standards. The o-xylene calibration standard concentrations must be at 5.0, 10, 50, 100, and 200 µg/L, while the concentration of the m- plus p-xylene isomers must total 5.0, 10, 50, 100, and 200 µg/L.

NOTE: The concentrations listed above are based on a 5 mL volume. If 10 mL volumes are to be used (i.e., low-level soil samples), then the concentrations of the standards must be reduced in half to ensure the same on-column amount of each analyte.

7.2.2.1.3 Calibration standards must be prepared in a volumetric flask or in the syringe used to inject the standard into the purging device.

7.2.2.1.4 For CCV (opening and closing CCVs), the standard shall be at the concentration equivalent to the mid-level calibration standards: 50 µg/L for non-ketones and 100 µg/L for ketones.

NOTE: The concentrations listed above are based on a 5.0 mL volume. If 10 mL volumes are to be used (i.e., low-level soil samples) then the concentrations of the standards must be reduced in half to ensure the same on-column amount of each analyte.

7.2.2.1.5 The methanol contained in each of the aqueous calibration standards must not exceed 1% by volume.

7.2.2.2 Initial Calibration Verification Solution

Prepare the working initial calibration standard solution containing all of the purgeable target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits) from an alternate source or a different lot than that used for the initial calibration (ICAL) standard analyses in methanol. Prepare a fresh calibration standard solution every month, or sooner if the solution has degraded or evaporated.

7.2.2.2.1 The ICV standard shall be at a concentration equivalent to the mid-level calibration standards: 50 µg/L for non-ketones and 100 µg/L for ketones.

7.2.2.2.2 The ICV standard shall be prepared by the same procedures as the CCVs.

7.2.2.3 Instrument Performance Check Solution

Prepare the instrument performance check solution containing BFB in methanol. If the BFB solution is added to the mid-level calibration standard (50 µg/L for non-ketones and 100 µg/L for ketones), add a sufficient amount of BFB to result in a 10 µg/L concentration of BFB (50 ng on-column). The BFB must be analyzed using the same GC and MS analytical conditions as are used for the calibration analysis.

7.2.2.4 Deuterated Monitoring Compound Spiking Solution

7.2.2.4.1 Prepare a DMC spiking solution in methanol (or in deuterated methanol) containing the compounds listed in Table 3 - Volatile Deuterated Monitoring Compounds and the Associated Target Analytes.

7.2.2.4.2 DMCs are to be added to each sample and blank, as well as initial calibration standards, ICV standard, and CCV standards.

7.2.2.4.3 For samples and blanks, add sufficient amount of the DMC spiking solution to each sample to result in the addition of 0.25 µg of each non-ketone DMC and 0.50 µg for each ketone DMC.

7.2.2.4.4 For ICAL, ICV, and CCV standards, add sufficient amounts of the DMC spiking solution to each 5.0 mL aliquot of calibration standard to result in the concentrations listed in Section 7.2.2.1.2 (initial calibration), Section 7.2.2.2.1 (ICV), and Section 7.2.2.1.4 (CCV).

7.2.2.4.5 Prepare a fresh DMC spiking solution every month, or sooner if the standard has degraded or concentrated.

7.2.2.5 Matrix Spiking Solution

If Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis is requested at the time of scheduling, prepare a spiking solution in methanol that contains the following analytes at a concentration of 12.5 µg/mL: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Prepare fresh spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.2.6 Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing 1,4-difluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4 in methanol. Add a sufficient amount of the internal standard spiking solution to samples, including MS/MSDs, blanks, and calibration standards to result in a 50 µg/L concentration or the addition of 0.25 µg for each internal standard. Prepare a fresh internal standard spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.3 Storage of Standard Solutions

7.2.3.1 Store the stock standards in PTFE-sealed screw-cap bottles with zero headspace at -10°C to -20°C.

7.2.3.2 Aqueous standards may be stored for up to 24 hours if held in PTFE-sealed screw-cap vials with zero headspace at ≤6°C, but not frozen. If not stored as such, the standards must be discarded after 1 hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be kept up to 12 hours in purge tubes connected via the autosampler to the P/T device.

7.2.3.3 Standard solutions purchased from a chemical supply company as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor that are immediately ampulated in glass vials may be retained for 2 years from the preparation date. The expiration date of the ampulated standards, upon the breaking of the glass seal, is 6 months (or sooner if the standard has degraded or evaporated).

7.2.3.4 Protect all standards from light.

7.2.3.5 Purgeable standards must be stored separately from other standards, samples, and blanks.

7.2.3.6 The Contractor is responsible for maintaining and verifying the integrity of standard solutions prior to use. Storage of standard solutions in the freezer may cause some compounds to precipitate. This means that standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in the solution.

7.2.3.6.1 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases may need to be replaced after 1 month for working standards and 6 months for opened stocks, or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

7.2.4 Temperature Records for Storage of Standards

7.2.4.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators/freezers.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Water Samples

8.1.1.1 Water samples may be collected in glass containers having a total volume of at least 40 mL with a PTFE-lined septum and an open top screw-cap.

8.1.1.2 The containers should have been filled in such a manner that no air bubbles were entrained to create a headspace in the vial.

8.1.1.3 The samples are preserved to a pH ≤2 at the time of collection.

8.1.1.4 A total of three vials per field sample is the recommended amount the Contractor should receive.

NOTE: If MS/MSD analysis is required for a particular sample, two additional vials should be sent by the field samplers. Contact the Sample Management Office (SMO) if insufficient sample for MS/MSD analysis has been provided.

8.1.2 Soil/Sediment Samples

8.1.2.1 Soil/Sediment samples may be received from the field either in pre-prepared closed-system P/T sample vials, pre-weighed glass vials, or in field core sampling/storage containers (e.g., EnCore™ or equivalent). Samples received in pre-prepared closed-system vials may arrive with no added preservatives, in 5 mL of water (low-level only), or preserved with sodium bisulfate (low-level only). Samples in pre-weighed glass vials may be preserved with 5 mL of methanol (medium-level samples only). Only vials that are thoroughly sealed may be used for medium-level soil analysis.

8.1.2.2 For soil samples received in pre-prepared, closed-system P/T sample vials (Section 10.2.2), or pre-weighed glass vials that are to be stored at -7°C, ensure that the samples are placed on their side prior to being frozen.

8.1.2.3 For samples received in pre-prepared closed-system P/T vials or pre-weighed glass vials, the Contractor should receive at least three such vials per field sample, plus at least one additional 60 mL sealed glass vial containing sample with minimum headspace. For samples received in field core sampling containers, the Contractor should receive at least three such containers per field sample, plus at least one additional 60 mL sealed glass vial containing sample with minimum headspace. If the minimum number of containers has not been sent by the field samplers, the Contractor is to immediately contact SMO for instructions. A total of four vials per field sample is the recommended amount of vials the Contractor should receive.

NOTE: If MS/MSD analysis is required for a particular sample, eight additional field core containers or glass vials should be sent by the field samplers. Contact SMO if insufficient sample for MS/MSD analysis has been provided.

8.1.2.3.1 For each methanol-preserved sample, samplers should send approximately 5 g (weight excluding preservative) of sample containing preservative in a pre-weighed glass vial. The Contractor shall weigh this vial immediately upon receipt and then store at ≤6°C, but not frozen. If a medium-level analysis of the sample is necessary, use this vial.

8.1.2.4 Samples received in pre-prepared closed-system P/T vials without preservative are to be stored at ≤6°C and analyzed within 24 hours of sample receipt, or they must be stored at less than -7°C until time of analysis if they do not contain visible moisture. If the sample appears to be moist and there is insufficient space above the soil portion in the sample vial, the Contractor shall contact SMO immediately for directions to avoid possible damage of the sample vial during sample storage in the freezer. Ensure that the samples are clean of external dirt and moisture prior to weighing.

8.1.2.5 In limited cases, preservation with sodium bisulfate may be required. Samples received in pre-prepared closed-system P/T vials preserved with sodium bisulfate shall be stored at ≤6°C, but not frozen, until time of analysis. Samples preserved with sodium bisulfate should be accompanied by field documentation recording the initial weight of the vial with preservative.

8.1.2.6 Medium-level samples may be received in pre-weighed vials preserved with methanol. Samples preserved with methanol should be accompanied by field documentation recording the initial weight of the vial with methanol. If the volume of methanol in the vial does not appear to be equal to 5 mL, or if the vial appears to be dry, or if field documentation of vial tare weight does not accompany the vials, the Contractor shall immediately contact SMO, who will contact the EPA Region.

8.1.2.7 For samples received in field core sampling/storage containers, the Contractor shall transfer the contents of the three containers for each sample, immediately upon receipt, to a pre-prepared closed-system P/T vial, and record the date and time of transfer. The transferred samples are to be analyzed within 24 hours of sample receipt, or they must be stored at less than -7°C. If the samples contain visible moisture, the Contractor shall immediately contact SMO.

8.2 Procedure for Sample and Sample Extract Storage

8.2.1 Sample Storage

8.2.1.1 Unpreserved low/medium soil samples must be protected from light and stored at less than -7°C from the time of receipt until time of analysis. Store unused sample aliquots at less than -7°C until 60 days after delivery of a complete reconciled, data package to the EPA. Samples received preserved with methanol shall be stored at ≤6°C, but not frozen, until time of analysis. Samples received without preservative are to be analyzed within 24 hours of sample receipt, or they must be stored at less than -7°C until time of preparation and analysis.

8.2.1.2 Sodium bisulfate preserved low soil samples and water samples must be protected from light and stored at ≤6°C, but not frozen, in a refrigerator used only for storage of volatile samples, in an atmosphere demonstrated to be free of all potential contaminants, until 60 days after delivery of a complete, reconciled data package to the EPA.

8.2.1.3 After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.1.4 Aqueous storage blanks shall be stored at ≤6°C, but not frozen, with preserved low/medium soil samples and water samples within an SDG until all such samples are analyzed. Inert sand storage blanks shall be stored at less than -7°C with unpreserved low/medium soil samples until all such samples are analyzed.

8.2.2 Sample Extract Storage

8.2.2.1 Medium level sample extracts must be protected from light and stored at -7°C until 365 days after delivery of a complete, reconciled data package to the EPA.

8.2.2.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3 Contract Required Holding Times

Analysis of water and soil/sediment samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). Analysis of unpreserved, unfrozen soil/sediment samples must be completed within 24 hours of VTSR. The holding time for the analysis of TCLP or SPLP filtrates and leachates is 7 days from the completion of the TCLP or SPLP filtration and extraction procedures.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Initial Instrument Set-up

9.1.1 Purge-and-Trap

9.1.1.1 The recommended P/T analytical conditions are provided in Table 5 – Purge and Trap Analytical Conditions. The conditions are suggested, but other conditions may be used, provided that all technical acceptance criteria are met for all standards, samples, and blanks.

9.1.1.2 Assemble a P/T device that meets the specifications in Section 6.3.4 and that is connected to a GC/MS system.

9.1.1.3 P/T instrumentation that allows internal standards and DMCs to be automatically added to each sample is widely available. Some of this instrumentation may be set up by the manufacturer to add only 1.0 µL of internal standard or DMCs. The 1.0 µL addition of standards will be allowed if the addition is done solely in an automated manner, and if the final concentration of the following standards in the 5 mL water samples and blanks can be met: 50 µg/L for internal standards; the concentrations listed in Section 7.2.2.1.2 for DMCs in the initial calibration; the concentrations listed in Section 7.2.2.2.1 for DMCs in the ICV; and the concentrations listed in Section 7.2.2.1.4 for DMCs in the CCV.

9.1.1.4 Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 mL/minute flow of inert gas according to the manufacturer’s recommendations. Do not vent the trap effluent onto the analytical column. Prior to daily use, condition the trap by heating at 180°C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be conditioned through the temperature program prior to the analysis of samples and blanks.

9.1.1.5 For low-level soil samples, establish the P/T instrument operating conditions. Adjust the instrument to inject 10 mL of reagent water, to heat the sample to 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer. Once established, the same P/T conditions must be used for the analysis of all standards, samples, and blanks.

9.1.1.6 Optimize P/T conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same P/T conditions must be used for the analysis of all standards, samples, and blanks.

NOTE: In certain situations, a heated purge may be used for water samples provided that all standards, samples, and blanks are analyzed under the same conditions and all technical acceptance criteria can be met.

9.1.1.7 A moisture reduction/water management system may be used to improve the chromatographic performance by controlling moisture if:

- The system does not introduce contaminants that interfere with identification and quantitation of target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated when using the moisture reduction/water management system meet the initial calibration, initial calibration verification, and continuing calibration verification technical acceptance criteria listed in the analytical method and the CRQLs listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits;
- All calibration standards, samples, and blanks are analyzed under the same conditions; and
- The Contractor performs acceptably on the Performance Evaluation (PE) samples using this system.

9.1.2 Gas Chromatograph

9.1.2.1 The recommended GC analytical conditions are provided in Table 6 – Gas Chromatograph Analytical Conditions. The conditions are recommended unless otherwise noted. GC conditions must achieve all performance criteria required for initial calibration, initial calibration verification, and continuing calibration.

9.1.2.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSDs.

9.1.2.3 Target analytes that are isomers (e.g., dichlorobenzenes) must be at least 50% resolved from each other. For xylene isomers, the two peaks representing o-xylene, m- and p-xylene, respectively, must be at least 50% resolved.

9.1.2.4 If the gaseous analytes chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shape, are not separated from the solvent front, or are not resolved greater than 90.0% from each other, then a subambient oven controller must be used, and the initial temperature must be less than or equal to 10°C.

9.1.3 Mass Spectrometer

The recommended MS analytical conditions are provided in Table 7 – Mass Spectrometer Analytical Conditions.

9.2 Instrument Performance Check

9.2.1 Summary of GC/MS Instrument Performance Check

9.2.1.1 The GC/MS system must be tuned to meet the manufacturer's specifications using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.2.3).

9.2.1.2 Prior to the analysis of any samples, including MS/MSDs, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing 4-BFB.

9.2.2 Frequency of GC/MS Instrument Performance Check

The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples, blanks, or standards are to be analyzed. The 12-hour period for the GC/MS instrument performance check, calibration standards (initial calibration, ICV, or CCV), blank, and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. However, in cases where a closing CCV can be used as an opening CCV for the next 12-hour period, then an additional BFB tune is not required, and the 12-hour period begins with the injection of the CCV. The time period ends after 12 hours have elapsed according to the system clock.

9.2.3 Procedure for GC/MS Instrument Performance Check

The analysis of the instrument performance check solution shall be performed as follows:

- As an injection of up to 50 ng of BFB into the GC/MS.
- By adding sufficient amount of BFB solution (Section 7.2.2.3) to 5.0 mL of reagent water to result in a 10 µg/L concentration of BFB.
- By adding a sufficient amount of BFB solution to the mid-level calibration standard to result in a 10 µg/L concentration of BFB.

9.2.4 Technical Acceptance Criteria for GC/MS Instrument Performance Check

9.2.4.1 The GC/MS system must be tuned at the frequency described in Section 9.2.2.

9.2.4.2 The abundance criteria listed in Table 2 – 4-Bromofluorobenzene Key Ions and Ion Abundance Criteria, must be met for a 50 ng injection of BFB. The mass spectrum of BFB must be acquired in the following manner:

9.2.4.2.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

9.2.4.2.2 Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of BFB. Do not background subtract part of the BFB peak.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a BFB analysis must be analyzed under identical GC/MS instrument analytical conditions.

9.2.5 Corrective Action for GC/MS Instrument Performance Check

9.2.5.1 If the BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source or take other corrective actions to achieve the technical acceptance criteria.

9.2.5.2 Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples (including MS/MSDs) and required blanks, and after the instrument performance check technical acceptance criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations (Section 7.2.2.1.2) to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target analytes and DMCs.

9.3.2 Frequency of Initial Calibration

9.3.2.1 Each GC/MS system must be calibrated prior to analyzing samples, whenever the Contractor takes corrective action which may change or affect the initial calibration criteria (e.g., ion source cleaning or repair, column replacement, etc.), or if the CCV acceptance criteria have not been met.

9.3.2.2 If time remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed (Section 9.3.5). It is not necessary to analyze another CCV standard. A method blank is required.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set up the GC/MS system as described in Section 9.1.

9.3.3.2 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.3.3.3 Add sufficient amount of the internal standard solution (Section 7.2.2.6) to each of the five aqueous calibration standard solutions (Section 7.2.2.1.2) containing the DMCs (Section 7.2.2.4.1) at the time of purge. Analyze each calibration standard according to Section 10.0 and outlined in Section 9.3.1. The initial calibration sequence is listed below.

INITIAL CALIBRATION SEQUENCE

1. GC/MS Instrument Performance Check
2. CS1 Initial Calibration Standard
3. CS2 Initial Calibration Standard
4. CS3 Initial Calibration Standard
5. CS4 Initial Calibration Standard
6. CS5 Initial Calibration Standard

9.3.3.4 Separate initial calibrations must be performed for water samples and low-level soil/sediment samples if different purge conditions are used (unheated purge vs. heated purge). Extracts of medium-level soil/sediment samples may be analyzed using the calibrations of water samples if the same purge conditions are used.

The Contractor may analyze different matrices in the same 12-hour period under the same tune, as long as separate calibration verifications are performed for each matrix within that 12-hour period.

9.3.4 Calculations for Initial Calibration

9.3.4.1 Calculate the RRF for each volatile target analyte and DMC using Equation 1. The primary characteristic ions used for quantitation are listed in Table 8 – Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards. If an interference prevents the use of a primary ion for a given internal standard, use a secondary ion listed in the same table. Assign the target analytes and DMCs to an internal standard according to Table 9 – Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

NOTE: Unless otherwise stated, the area response of the primary characteristic ion is the quantitation ion.

EQ. 1 Relative Response Factor

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

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (Table 8 - Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (Table 8 - Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards). The target analytes are listed with their associated internal standards in Table 9 – Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

C_{is} = Concentration or amount of the internal standard

C_x = Concentration or amount of the analyte to be measured

9.3.4.2 Calculating the RRFs of the xylenes requires special attention. Report an RRF for m,p-xylene and one for o-xylene. On the available capillary columns, the m,p-xylene isomers coelute. Therefore, when calculating the RRF in the equation above, use the area response (A_X) and concentration (C_X) of the peak from o-xylene, and A_X and C_X of the peak from m,p-xylene isomers respectively.

9.3.4.3 The Mean RRF ($\overline{RRF}$) must be calculated for all compounds according to Equation 2.

9.3.4.4 Calculate the Percent Relative Standard Deviation (%RSD) of the RRF values for each purgeable target analyte and DMC over the initial calibration range using Equation 3 in conjunction with Equations 2 and 4.

9.3.4.4.1 Equation 2 is the general formula for the mean of a set of values.

EQ. 2 Mean Value

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

WHERE,

X_i = Value

$\overline{X}$ = Mean value

n = Number of values

9.3.4.4.2 Equation 3 is the general formula for the relative standard deviation.

EQ. 3 Percent Relative Standard Deviation

$$\%RSD = \frac{SD_{RRF}}{\overline{X}} \times 100$$

WHERE,

SD_{RRF} = Standard deviation of initial calibration RRFs (per compound) from EQ. 4

$\overline{X}$ = Mean value of the initial calibration RRFs (per compound)

9.3.4.4.3 Equation 4 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 4 Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{(n-1)}}$$

WHERE,

X_i = Each individual value used to calculate the mean

$\overline{X}$ = The mean of n values

n = Total number of values

9.3.5 Technical Acceptance Criteria for Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentrations described in Section 7.2.2.1.2, and at the frequency described in Section 9.3.2 on a GC/MS system meeting the BFB technical acceptance criteria (Section 9.2.4).

9.3.5.2 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.3.5.3 The RRF at each calibration concentration for each target analyte and DMC that has a required minimum RRF value must be greater than or equal to the compound's minimum acceptable RRF listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds.

9.3.5.4 The %RSD for each target analyte or DMC listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds must be less than or equal to the value listed.

9.3.5.5 Up to two target analytes and DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds.

9.3.5.6 Up to two target analytes and DMCs (excluding those with maximum %RSD requirements of 40.0%) may fail to meet the criteria listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds, but these compounds must still meet the maximum %RSD requirements of 40.0%.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, service the P/T device, or take other corrective actions to achieve the technical acceptance criteria.

9.3.6.2 It may be necessary to adjust the purge gas (helium) flow rate (normally in the range of 25-40 mL/minute). Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

9.3.6.3 Any samples or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.4 Initial Calibration Verification

9.4.1 Summary of Initial Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check and initial calibration technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an ICV (containing all the purgeable target analytes from an alternate source or a different lot than the ICAL standards, and the DMCs and internal standards from the same source or lot as in the ICAL standards) to ensure that the instrument is calibrated accurately.

9.4.2 Frequency of Initial Calibration Verification

The calibration for each GC/MS system used for analysis must be verified with an ICV at the frequency of one per ICAL analytical sequence. The ICV shall be analyzed following that last ICAL standard analysis and prior to any method blank, sample, or applicable CCV analysis.

Injection #	Material Injected
1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	BFB then CS1–CS5 First 6 steps of the initial calibration
7th - ICV	ICV
8th - blanks, samples, MS/MSDs	Blanks, samples, and MS/MSDs
9th - Subsequent Samples	

9.4.3 Procedure for Initial Calibration Verification

9.4.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.4.3.2 Add sufficient amount of internal standard solution (Section 7.2.2.6) to the ICV (Section 7.2.2.2) and the DMC solution (Section 7.2.2.4). Analyze the ICV Standard according to Section 10.0.

9.4.3.3 For low-level soil samples, the ICV standard shall be prepared in the same manner as the initial calibration standard of the same concentration as specified in Section 7.2.2.1.

9.4.4 Calculations for Initial Calibration Verification

9.4.4.1 Calculate an RRF for each target analyte and DMC according to Section 9.3.4.1.

9.4.4.2 Calculate the Percent Difference (%D) between the ICV RRF_c and the preceding initial calibration $\overline{RRF_i}$ for each purgeable target analyte and DMC using the following equation:

EQ. 5 Initial Calibration Verification Percent Difference

$$\%D = \frac{RRF_C - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

WHERE,

RRF_c = Relative Response Factor from current ICV standard

$\overline{RRF_i}$ = Mean Relative Response Factor from the preceding initial calibration

9.4.5 Technical Acceptance Criteria for Initial Calibration Verification

9.4.5.1 The concentration of the low/medium volatile organic target analytes and DMCs in the ICV must be at or near the mid-point concentration of the calibration standards (50 µg/L for non-ketones and 100 µg/L for ketones). The ICV must be analyzed at the frequency described in Section 9.4.2, on a GC/MS system meeting the BFB (Section 9.2.4) and the initial calibration (Section 9.3.5) technical acceptance criteria.

9.4.5.2 For an ICV, the RRF for each target analyte and DMC must be greater than, or equal to, the compound's minimum RRF listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds. Up to two target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds, but these compounds must still meet the minimum RRF requirements of 0.010.

9.4.5.3 For an ICV, the %D for each target analyte and DMC listed in Table 4 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds must be in the inclusive range of the compound's %D values listed.

9.4.5.4 No quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Initial Calibration Verification

9.4.6.1 If the ICV technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3.

9.4.6.2 If the ICV fails to meet the technical acceptance criteria and a subsequent reanalysis of the ICV meets the technical acceptance criteria, proceed to the blank and sample analyses. All sample and required blank analyses must be associated to a compliant ICV analysis following the associated ICAL.

9.5 Continuing Calibration Verification

9.5.1 Summary of Continuing Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check, initial calibration, and ICV technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an opening CCV (containing all the purgeable target analytes, DMCs, and internal standards) to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the analytical method. A closing CCV using the same standard conditions as for the opening CCV is required after all samples and blanks have been analyzed, and before the end of the 12-hour period (refer to the analytical sequence in Section 9.5.2.3).

9.5.2 Frequency of Continuing Calibration Verification

9.5.2.1 The calibration for each GC/MS system used for analysis must be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the injection of BFB, followed by the injection of the opening CCV solution. BFB may be added to the CCV solution, in which case only one injection is necessary. If a closing CCV meets the technical acceptance criteria for an opening CCV (Section 9.5.5) and samples are analyzed within that subsequent 12-hour period, then an additional BFB tune is not required and the 12-hour period begins with that calibration verification. If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then a BFB tune, followed by an opening CCV, is required and the next 12-hour period begins with the BFB tune (Section 9.2.2).

9.5.2.2 If time remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration and ICV, samples may be analyzed. A method blank is required.

9.5.2.3 After the injection of all samples and required blanks, and before the end of the 12-hour period, another injection of the CCV solution is required (closing CCV). The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence, provided that all technical acceptance criteria are met for an opening CCV in Section 9.5.5.

Time	Injection #	Material Injected
0 hr	1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	BFB then CS1–CS5 First 6 steps of the initial calibration
	7th - ICV	ICV
	8th - blanks, samples, MS/MSDs	Blanks, samples, and MS/MSDs
	9th – Subsequent Samples	
End 12 hr	Closing CCV (meeting Closing CCV criteria but not Opening CCV)	CS3 – Closing CCV
New 12 hr	1st GC/MS Instrument Performance Check	BFB Instrument Performance Check
	2nd - Analysis past 12 hours Opening CCV	CS3 - Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
End 12 hr	Closing CCV (meeting Closing CCV criteria but not Opening CCV)	CS3 – Closing CCV
New 12 hr	1st Analysis Instrument Performance Check	BFB Instrument Performance Check
	2nd Analysis Opening CCV	CS3 – Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
		Storage Blank if previous sample is the last sample in SDG
End of 12 hr beginning of next 12 hr	Closing CCV (meeting Opening CCV criteria) Instrument Performance Check not required	CS3 – Closing CCV meeting Opening CCV
		Blank, MS/MSD, subsequent samples
		Subsequent Samples
		Last Sample
		Storage Blank (after last sample in SDG)
End of 12 hr	Closing CCV meeting criteria	CS3 – Closing CCV meeting Opening CCV

9.5.3 Procedure for Continuing Calibration Verification

9.5.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.5.3.2 Add a sufficient amount of the internal standard solution (Section 7.2.2.6) to the CCV (Section 7.2.2.1.4) and the DMC solution (Section 7.2.2.4). Analyze the CCV standard according to Section 10.0.

9.5.3.3 For low-level soil samples, the CCV standard shall be prepared in the same manner as the initial calibration standard of the same concentration as specified in Section 7.2.2.1.

9.5.4 Calculations for Continuing Calibration Verification

9.5.4.1 Calculate an RRF for each target analyte and DMC according to Section 9.3.4.1.

9.5.4.2 Calculate the %D between the CCV RRF_C and the most recent initial calibration $\overline{RRF_i}$ for each purgeable target analyte and DMC using the following equation:

EQ. 6 Internal Standard Calibration Percent Difference

$$\%D = \frac{RRF_C - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

WHERE,

RRF_C = Relative Response Factor from current CCV standard

$\overline{RRF_i}$ = Mean Relative Response Factor from the most recent initial calibration

9.5.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.5.5.1 The concentration of the low/medium volatile organic target analytes and DMCs in the opening and closing CCV must be at or near the mid-point concentration of the calibration standards (50 µg/L for non-ketones and 100 µg/L for ketones). The opening and closing CCV must be analyzed at the frequency described in Section 9.5.2, on a GC/MS system meeting the BFB (Section 9.2.4), initial calibration (Section 9.3.5), and the ICV (Section 9.4.5) technical acceptance criteria.

9.5.5.2 For an opening or closing CCV, the RRF for each target analyte and DMC must be greater than, or equal to, the compound's opening or closing minimum RRF listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds.

9.5.5.3 For an opening CCV, the %D for each target analyte and DMC listed in Table 4 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Volatile Organic Compounds must be in the inclusive range of the compound's %D values listed. For a closing CCV, the %D for each target analyte and DMC must be in the inclusive range of the compound's %D value listed. Up to two target analytes and/or DMCs in the closing CCV are allowed to exceed the %D values listed.

9.5.5.4 For an opening or closing CCV, up to two target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Section 9.5.5.2, but these compounds must still meet the minimum RRF requirements of 0.010.

9.5.5.5 No quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.5.6 Corrective Action for Continuing Calibration Verification

9.5.6.1 If the opening CCV technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3. If the closing CCV technical acceptance criteria are not met, then all samples and blanks analyzed within that 12-hour period must be reanalyzed at no additional cost to the EPA.

9.5.6.2 The Contractor shall follow the procedure in Section 10.2.4.1 if they cannot meet the control criteria after the analysis of an original undiluted or minimally diluted sample due to matrix interference. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the CCV technical acceptance criteria.

9.5.6.3 Any samples or required blanks analyzed when opening CCV technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.5.6.4 The corrective action for sample reanalysis is not required when noncompliant analytes or associated DMCs, in the opening or closing CCVs bracketing a dilution or a reanalysis, are not the same analytes or associated DMCs for which the dilution analysis or reanalysis was intended.

10.0 PROCEDURE

10.1 Introduction to Sample Analysis

Samples shall be analyzed only after the GC/MS system has met the technical requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration. All samples, required blanks, and standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

NOTE: Contact SMO if sample vials have bubbles entrained resulting in headspace.

10.2 Procedure for Sample Analysis

10.2.1 Water Samples

10.2.1.1 If time remains in the 12-hour period (as described in Section 9.2.2), samples may be analyzed without analysis of a CCV standard.

10.2.1.2 If the autosampler can automatically sample the appropriate volume, then the following Sections 10.2.1.3 - 10.2.1.6 are performed by the autosampler.

10.2.1.3 Remove the plunger from a 5.0 mL syringe and attach a closed syringe valve. Open the sample or standard bottle that has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Invert the

syringe, open the syringe valve, and vent any residual air while adjusting the sample volume to 5.0 mL.

10.2.1.4 This process of taking an aliquot destroys the validity of the sample for future analysis, unless the excess sample is immediately transferred to a smaller vial with zero headspace and stored at ≤6°C, but not frozen. Therefore, if only one sample vial is provided, the analyst must fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly. Filling one 5.0 mL syringe would allow only one analysis of that sample. If an analysis is needed from the second 5.0 mL syringe, it must be performed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

10.2.1.5 Add a sufficient amount of DMC spiking solution (Section 7.2.2.4) and a sufficient amount of internal standard spiking solution (Section 7.2.2.6) through the valve bore of the syringe, then close the valve. Invert the syringe 3 times. The DMCs and internal standards may be mixed and added as a single spiking solution.

10.2.1.6 Once the sample aliquots have been taken from the VOA vial, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do **not** add pH paper to the vial). Record the pH of each sample and report these data in the SDG Narrative, following the instructions in Exhibit B – Reporting and Deliverables Requirements. No pH adjustment is to be performed by the Contractor.

10.2.1.7 Attach the valve assembly on the syringe to the valve on the sample sparger. Open the valves and inject the sample into the purging chamber.

10.2.1.8 Close both valves and purge the sample under the same conditions as the initial calibration.

10.2.1.9 Sample Desorption - After the purge is complete, attach the trap to the GC, adjust the P/T system to desorb mode, initiate the temperature program sequence of the GC, and start data acquisition. Introduce the trapped material into the GC column by rapidly heating the trap to the appropriate desorb temperature while backflushing the trap with inert gas. While the trapped material is being introduced into the GC, empty the sample sparger and rinse it with reagent water. 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 sample sparger with a detergent solution, rinse it with reagent water, and then dry it in an oven at 105°C.

10.2.1.10 Trap Reconditioning - After desorbing the sample, recondition the trap in accordance with manufacturer's instructions with the recommended trap recondition for a minimum of 7.0 (±0.1) minutes at 180°C. The same conditions must be used for all analyses.

10.2.1.11 Termination of Data Acquisition - 3 minutes after all the purgeable target analytes have eluted from the GC, terminate the MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and appropriate EICPs.

10.2.2 Low-Level Soil/Sediment Samples

10.2.2.1 If samples are received as sealed VOA vials or containers, they are to be analyzed according to Section 10.2.2.9, unless screening analysis indicates samples should be analyzed as medium-level samples. If the results of medium-level analysis indicate that all target analyte concentrations are below the medium-level CRQL in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, then the samples must be analyzed as low-level samples. If samples are originally analyzed by the low-level method, and any target analyte in the sample exceeds the concentration of the same target analyte in the high standard, then the sample may be analyzed at a dilution per Section 10.2.4, or by the medium-level method (Section 10.2.3). If the laboratory suspects that any target analyte is at a concentration that may result in instrument performance problems when analyzed even using the medium-level method, SMO should be contacted for further guidance.

If the EPA specifically requests the laboratory to analyze a sample only by the medium-level protocol (i.e., methanol extraction technique), the laboratory is not obligated to perform the low-level analysis. The request to the laboratory is to be made on the Traffic Report/Chain of Custody (TR/COC) Record. After receiving a TR/COC Record with this specific request, the laboratory is to confirm the request through SMO.

10.2.2.2 The following steps apply to the preparation of vials used for the analysis of low-level soil/sediment samples by the closed-system P/T equipment described in this method. If samples are not received in closed-system purge vials, proceed to Section 10.2.2.7.

NOTE: There should be three field core sampling/storage containers for each field sample. The contents of two of the field core containers are to be transferred immediately upon sample receipt and processed using the steps outlined in Sections 10.2.2.9 - 10.2.2.10. One of these prepared samples is then to be used as the primary sample, while the other is to be used as a back-up sample, if necessary. The contents of the third field core container shall be transferred immediately upon sample receipt to a tared dry closed-system P/T container (i.e., no preservative solution or stirring bar is to be added), weighed according to Section 10.2.2.8, and then stored at less than -7°C. This sample shall be used for the medium concentration level methanol extraction procedure as described in Section 10.2.3, if results of the original analysis indicate that medium-level extraction is warranted.

10.2.2.3 Add a clean magnetic stirring bar to each clean vial. If the P/T device stirs the sample with a means other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

10.2.2.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.

10.2.2.5 Affix a label to each vial and weigh the prepared vial to the nearest 0.01 g. Record the tare weight and final weight.

10.2.2.6 Because volatile organics will partition into the headspace of the vial and will be lost when the vial is opened, DMCs, MS/MSDs, and internal standard spiking solutions should only be added to vials after the sample has been added to the vial. The spiking solutions should be introduced either manually by puncturing the septum with a small-gauge needle or automatically by the P/T system just prior to analysis.

10.2.2.7 Using the sample collection device, transfer the contents (approximately 5 g) into the sample vial. This sample transfer must be performed rapidly to minimize loss of volatile analytes. Quickly brush any soil off the vial and immediately seal the vial with the septum and screw-cap. The soil vial is hermetically sealed and must remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. Record the date and time of sample transfer to pre-prepared vials and also submit this information with the data package.

10.2.2.8 Weigh the vial and contents to the nearest 0.01 g and record this weight. Sample weight is determined by subtracting the sample vial tared weight (Section 10.2.2.5) from this final weight. For samples received in closed-system purge vials, the tared weights should have been provided by the field sampler. If tared weights are not provided, contact SMO for further guidance.

10.2.2.9 Prior to sample purge, all soil/sediment samples must be allowed to warm to ambient temperature. All low-level soil samples should have a total sample volume (reagent water and preservative) of 10 mL. For those samples that have been stored in freezing compartments and will be analyzed by the low concentration level protocol, 10 mL of reagent water must be added to the vials without disturbing the hermetic seal of the sample vial.

For preserved samples, add additional 5.0 mL of reagent water to the vial.

Shake all vials containing aqueous solutions gently to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer’s instructions.

10.2.2.10 Without disturbing the hermetic seal on the sample vial, add 5.0 mL reagent water, sufficient amount of the internal standard spiking solution (Section 7.2.2.6), and the DMC spiking solution (Section 7.2.2.4). All samples, including MS/MSDs, standards, and blanks, within an SDG must have the same amount of reagent water added. Do not increase/change the amount of DMC and internal standard solution added.

10.2.2.11 Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

10.2.2.12 Purge the sample under the same conditions as the initial calibration, while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

10.2.2.13 If a non-cryogenic interface is to be utilized, place the P/T system in the desorb mode after the purge interval, and preheat the trap to the desorb temperature without a flow of desorption gas. Start the flow of desorption gas. Begin the temperature program of the GC and start data acquisition.

10.2.2.14 If a cryogenic interface is to be utilized, place the P/T system in the desorb mode after the purge interval, making sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to desorb the sample. At the end of the desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the GC and start the data acquisition.

10.2.2.15 After desorbing the sample, recondition the trap and adjust the P/T system to prepare for the next sample.

10.2.3 Medium-Level Soil/Sediment Samples

10.2.3.1 The medium-level soil/sediment method is based on extracting the soil/sediment sample with methanol. An aliquot of the methanol extract is added to reagent water containing the DMCs and the internal standards. The reagent water containing the methanol extract is purged at ambient temperature.

10.2.3.2 Prior to the analysis of samples, establish the appropriate P/T GC/MS operating conditions, as outlined in Section 9.1.1. Because the methanol extract and reagent water mixture is purged at ambient temperature, the instrument performance check, initial calibration, and CCV for water samples shall be used for analyses of medium-level soil/sediment sample extracts.

10.2.3.3 Weigh the vial and contents to the nearest 0.01 g and record this weight. Sample weight is determined by subtracting the sample vial tared weight determined in Section 10.2.2.5. For samples received in closed-system purge vials, the tared weights should have been provided by the field sampler. If tared weights are not provided, contact SMO for further guidance.

NOTE: If a methanol preserved sample is to be analyzed, weigh the sample vial and contents to the nearest 0.01 g and record the weight. Record any discrepancies between laboratory-determined weight and sampler-determined weight in the SDG Narrative and utilize the sampler-determined weight in any calculations. Proceed to Section 10.2.3.5.

10.2.3.4 Quickly add 5.0 mL of methanol to the vial. Cap and shake for 2 minutes.

NOTE: The steps in Sections 10.2.3.3 and 10.2.3.4 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

10.2.3.5 Let the solution settle. Then, using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be discarded. The 1 mL extract may be stored in the dark at ≤6°C, but not frozen, prior to the analysis.

10.2.3.6 Add 100 µL of the methanol extract to the 4.9 mL of reagent water for analysis. Otherwise, estimate the concentration range of the sample from the low-level analysis or from the in-house screening procedure to determine the appropriate volume. A 100 µL of methanol extract is the maximum volume that can be added to the 4.9 mL of reagent water for medium-level analysis. If less than

100 µL of methanol extract is used, a volume of clean methanol must be used so that the volumes of methanol extract and clean methanol total 100 µL.

10.2.3.7 Remove the plunger from a 5.0 mL Luer-Lok type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5 mL to allow volume for the addition of sample and standards, and add sufficient amount of DMC spiking solution (Section 7.2.2.4) and sufficient amount of internal standard spiking solution (Section 7.2.2.6). Also add the volume of methanol extract determined in Section 10.2.3.6 and a volume of clean methanol (if necessary) to total 100 µL (excluding methanol in the DMC/internal standard spiking solutions).

10.2.3.8 Attach the syringe-syringe valve assembly to the syringe valve on the purge device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

10.2.3.9 Proceed with the analysis as outlined in Sections 10.2.2.12 - 10.2.2.15.

10.2.4 Sample Dilutions

10.2.4.1 The Contractor shall analyze samples undiluted, or at minimal dilution. Samples may be diluted because of target analyte concentration exceeding the concentration of the same target analyte in the high standard, or because of excessive matrix interference that hinders accurate quantitation. It is highly recommended that screening analysis be performed prior to sample analysis to determine estimated analyte concentration and matrix problems.

NOTE 1: If the laboratory has evidence or highly suspects, because of sample color or other physical properties, that a sample may contain high concentrations of either target or non-target analytes, then the Contractor shall contact SMO to obtain guidance from the EPA as to whether a smaller aliquot or the medium-level method (Section 10.2.3) would be most appropriate.

NOTE 2: In the event that interference precludes accurate quantitation using the primary quantitation ion, but a secondary ion with less interference could be used instead, then secondary ion quantitation should be considered (see Section 11.2.1.4).

10.2.4.2 For water samples, samples may be diluted to keep target analyte concentrations within the calibrated range and/or to keep baseline height from the earliest eluting peak from exceeding one-half the relative height of the highest peak in the chromatogram. If dilution is required due to baseline drift, the laboratory must submit chromatograms in which the highest peak is set to full scale. If the baseline rises less than 10% in the diluted analysis, the sample has been overdiluted.

10.2.4.3 For soil samples analyzed by the low-level method, if the concentration of any target analyte in the sample exceeds the concentration of the same target analyte in the high standard, then the Contractor shall proceed with the medium level sample analysis.

10.2.4.4 Samples may be diluted in a volumetric flask or in a 25 mL Luer-Lok syringe.

10.2.4.5 The Dilution Factor (DF) chosen must keep the concentrations of the volatile target analytes that required dilution in the upper half of the calibration range.

10.2.4.6 All dilutions must be made just prior to GC/MS analysis of the sample. Until the diluted sample is in a gas-tight syringe, all steps in the dilution procedure must be performed without delay.

10.2.4.7 To dilute the sample in a volumetric flask, use the following procedure:

10.2.4.7.1 Select the volumetric flask that will allow for the necessary dilution (10-100 ml). Intermediate dilution may be necessary for extremely large dilutions.

10.2.4.7.2 Calculate the approximate volume of appropriately acidified reagent water that will be added to the selected volumetric flask and add slightly less than this quantity of the reagent water to the flask.

10.2.4.7.3 For water samples, inject the proper aliquot from the syringe into the volumetric flask. Only aliquots of 1.0 mL increments are permitted. Dilute the aliquot to the mark on the flask with reagent water. Cap the flask and invert it 3 times.

10.2.4.7.4 Fill a 5.0 mL syringe with the diluted sample as in Section 10.2.1.3. If this is an intermediate dilution, use it and repeat the above procedure to achieve larger dilutions.

10.2.4.8 If more than two analyses (i.e., from the original sample and more than one dilution, or from the most concentrated dilution analyzed and further dilutions) are required to get concentrations of all target analytes within the calibration range, contact SMO.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Analytes

11.1.1.1 The analytes listed in the TAL in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of the standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component within the Gas Chromatographic Relative Retention Time (RRT) unit window established from the 12-hour calibration standard; and
- Correspondence of the sample component and calibration standard analyte mass spectra.

11.1.1.2 For establishing correspondence of the GC RRT, the sample component RRT must be within ±0.06 RRT units of the RRT of the corresponding continuing calibration standard component. For reference, the standard must be analyzed in the same 12-hour period as the sample. If samples are analyzed during the same 12-hour period as the initial calibration standards, use the RRT values from the 50 µg/L standard. Otherwise, use the corresponding opening CCV standard. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, then the RRT should be assigned by using EICPs for ions unique to the component of interest.

11.1.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS (as opposed to library spectra) are required. Once obtained, these standard spectra may be used for identification purposes, only if the Contractor's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the standard analysis that was also used to obtain the RRTs.

11.1.1.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:

11.1.1.4.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.1.1.4.2 The relative intensities of ions specified in the section above must agree within ±20% between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30-70%).

11.1.1.4.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra.

11.1.1.4.4 If an analyte cannot be verified by all of the spectral identification criteria listed in Section 11.1.1.4, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the Contractor shall report that identification and proceed with quantitation and document in the SDG Narrative.

11.1.2 Identification of Non-Target Compounds

11.1.2.1 A library search shall be executed for non-target compounds for the purpose of tentative identification. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library, shall be used as the reference library.

11.1.2.2 All organic compounds that have not been positively identified as volatile target analytes using the procedures detailed in Section 11.1.1, or that are not DMCs, internal standards, or semivolatile target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, shall be tentatively identified via a forward search of NIST, Wiley, or equivalent mass spectral library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer-generated library search routines must not use normalizations which would misrepresent the library or unknown spectra when compared to each other.

11.1.2.3 Up to 30 non-alkane Tentatively Identified Compounds (TICs) of greatest apparent concentration shall be reported on Form 1B-OR. Peaks that are tentatively identified as straight-chain, branched, or cyclic alkanes, and are alone or part of an alkane series, shall be reported as "total alkanes". An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The concentrations of each of the alkanes are to be summed and reported as a single result for the "total alkanes". The alkanes are not to be counted as part of the 30 compounds individually reported as TICs on Form 1B-OR. Carbon dioxide and compounds with responses less than 10% of the internal standard with which they are to be quantified (as determined by inspection of the peak areas or height) are not to be reported (nor are they to be counted as part of the 30 compounds that are to be reported).

11.1.2.4 Rules for Making Tentative Identification

11.1.2.4.1 For compounds to be reported, as per the instructions in Section 11.1.2, identification (as generated by the library search program) of those receiving a library search match of 85% or higher should be considered a "probable match". The compound should be reported with the identification generated by the search program, unless the mass spectral interpretation specialist feels there is just evidence not to report the compound as identified by the library search program.

11.1.2.4.2 If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match. Do not report DMCs, internal standards, or analytes that are on the volatile

or semivolatile TAL, unless semivolatile analysis is not being done.

11.1.2.4.3 If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethylnaphthalenes), the compound with the highest library search percent match should be reported (or first compound if library search matches are the same).

11.1.2.4.4 If the mass spectral interpretation specialist has just evidence to support reporting a compound with a tentative identification of something other than that generated by the library search program (with a library search result of 85% or greater), the laboratory shall include in the SDG Narrative the justification for not reporting a compound as listed by the search program. This narrative shall detail explicitly why a library search generated identification for a compound was rejected. If a TIC has obvious isomer analogs, the laboratory shall include in the SDG Narrative a statement indicating that the exact isomer configuration, as reported, may not be absolutely accurate.

11.1.2.4.5 If the library search produces no matches at or above 85%, the mass spectral interpretation specialists are encouraged to make a valid tentative identification of the compound. If no valid tentative identification can be made, the compound should be reported as "unknown". The mass spectral interpretation specialist should give additional classification of the unknown, if possible (e.g., "unknown aromatic compound", "unknown chlorinated compound", etc.).

11.1.2.4.6 The Chemical Abstracts Service (CAS) registry number is the unique identifier for each chemical compound. As the rules of chemical nomenclature have changed over time, each chemical substance is liable to have several names or synonyms: trade or brand name(s); generic or common name(s); trivial or systematic; or International Union of Pure and Applied Chemistry (IUPAC) name(s). Whether synonyms or other names are created for this compound, the CAS registry number will generally remain unchanged. The CAS registry number is simply an identifier which has no structural significance. Regardless of retention times (RTs), if the library search produces two or more compounds, at or above 85% with the same Chemical Abstract Number, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds) unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match.

11.1.2.4.7 If the library search produces only one and the same compound (i.e., the same CAS registry number) with percent match at or above 85% at two different RTs, the compound having the highest percent match should be reported as TIC and the other one could be reported as unknown. If both TICs have the same percent match for the same compound, one of the TICs could be reported as unknown. Such justifications should be included in the SDG Narrative.

11.2 Quantitative Analysis

11.2.1 Data Processing Procedure

11.2.1.1 Target analytes identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (Table 9 – Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation). The EICP area of primary characteristic ions of analytes listed in Table 8 – Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards, are used for quantitation.

11.2.1.2 For water, low-level soil/sediment samples, and medium-level soil/sediment samples, xylenes are to be reported as "m,p-xylenes" and "o-xylene". Because m- and p-xylene isomers coelute, special attention must be given to the quantitation of the xylenes. In quantitating sample concentrations, be sure to use the correct corresponding RRF values.

NOTE: The area of each peak (i.e., the peaks for o-xylene and m,p-xylene) must appear on the quantitation report.

11.2.1.3 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene are to be reported separately.

11.2.1.4 Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion, not when saturation occurs. If secondary ion quantitation is used, calculate an RRF using the area response (EICP) from the most intense secondary ion which is free of sample interferences, and document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

11.2.1.5 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitation. This normally occurs when there is compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific target analyte, DMC, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall also not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instances of manual integration must be documented in the SDG Narrative.

11.2.1.6 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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 instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the

quantitation ion displaying the manual integration(s). This applies to all target analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

11.2.2 Target Analyte Calculations

11.2.2.1 Identified target analytes shall be quantitated by the internal standard method using Equation 7, 8 or 9. The internal standard used shall be that which is assigned in Table 9 - Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation. The $\overline{RRF}$ from the initial calibration standard is used to calculate the concentration in the sample.

11.2.2.2 Water

EQ. 7 Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_{is})(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and DMCs are listed in Table 8 – Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The primary quantitation ions for the internal standards are in Table 8 – Characteristic Ions for Volatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards. The target analytes are listed with their associated internal standards in Table 9 - Volatile Target Analytes and Deuterated Monitoring Compounds with Associated Internal Standards for Quantitation.

I_{is} = Amount of internal standard added, in ng

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration standard

V_o = Total volume of water purged, in mL

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5.0 mL with reagent water and purged, DF = 5.0 mL/2.0 mL = 2.5. If no dilution is performed, DF = 1.0.

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated concentration by 1000.

11.2.2.3 Low-Level Soil/Sediment

EQ. 8 Low-Level Soil/Sediment Concentration

$$\text{Concentration } (\mu g/Kg) = \frac{(A_x)(I_{is})(DF)}{(A_{is})(\overline{RRF})(W_s)(S)}$$

WHERE,

A_x, I_{is}, A_{is}, DF = As given for water, EQ. 7

$\overline{RRF}$ = Mean Relative Response Factor from the heated purge of the initial calibration standard

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

W_s = Weight of sample added to the purge tube, in g

11.2.2.4 Medium-Level Soil/Sediment

EQ. 9 Medium-Level Soil/Sediment Concentration

$$\text{Concentration}(\mu g/Kg) = \frac{(A_x)(I_{is})(AV_t)(1000)(DF)}{(A_{is})(\overline{RRF})(V_a)(W_s)(S)}$$

WHERE,

A_x, I_{is}, A_{is} = As given for water, EQ. 7

S = As given in EQ. 8

$\overline{RRF}$ = Mean Relative Response Factor from the ambient temperature purge of the initial calibration standard

AV_t = Adjusted total volume of the methanol extract plus soil water in mL determined by:

$AV_t = V_t + \{W_s - [W_s(S)]\}$

Where V_t = total volume of methanol extract in mL. This volume is typically 5.0 mL, even though only 0.1 mL is transferred to the vial in Section 10.2.3.6. The quantity derived from $\{W_s - [W_s(S)]\}$ is the soil water volume and is expressed in mL.

V_a = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 µL), in µL added to reagent water for purging

W_s = Weight of soil/sediment extracted, in g

DF = Dilution Factor. The DF for analysis of soil/sediment sample extracts for volatiles by the medium-level method is defined as the ratio of the volume (µL) taken from the extract used to make the dilution plus the clean solvent added for the dilution (µL), to the volume taken from the extract used to make the dilution. For example, if 10 µL of the extract was taken and added to 90 µL of clean solvent, then ration would be (10 µL + 90 µL/10 µL)= a DF of 10.

11.2.3 Non-Target Compounds

11.2.3.1 An estimated concentration for TICs shall be determined by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used.

11.2.3.2 Equations 7, 8, and 9 are also used for calculating TIC concentrations. Total area counts (or peak heights) from the total RICs are to be used for both the TIC to be measured (A_x) and the internal standard (A_{is}). An $\overline{RRF}$ of 1.0 is to be assumed.

11.2.4 Contract Required Quantitation Limit Calculations

11.2.4.1 Water

EQ. 10 Water and TCLP/SPLP Leachate Sample Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{V_c}{V_o} \times DF$$

WHERE,

Contract CRQL = CRQL value reported in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits

V_o, DF = As given in EQ. 7

V_C = Method required purge volume

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated CRQL by 1000.

11.2.4.2 Low-Level Soil/Sediment

EQ. 11 Low-Level Soil Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_c)}{(W_s)\,(S)}$$

WHERE,

W_s, S = As given in EQ. 8

W_C = Method required sample weight (5.0 g)

11.2.4.3 Medium-Level Soil/Sediment

EQ. 12 Medium-Level Soil/Sediment Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL X} \frac{(W_x)\,(AV_t)\,(V_y)\,(1000)\,(DF)}{(W_s)\,(V_c)\,(V_a)\,(S)}$$

WHERE,

AV_t, DF, W_s, V_a, S = As given in EQ. 9

W_x = Method required sample weight (5.0 g)

V_y = Method required soil aliquot volume from soil methanol extract (100 µL)

V_c = Method required soil methanol extract volume (5,000 µL)

11.2.5 Deuterated Monitoring Compound Recoveries

11.2.5.1 Calculate the concentration of each DMC using the same equation as used for target analytes.

11.2.5.2 Calculate the recovery of each DMC in all samples and blanks using Equation 13. Report the recoveries on the appropriate forms.

EQ. 13 DMC Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

WHERE,

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

11.3 Technical Acceptance Criteria for Sample Analysis

11.3.1 The samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, CCV, and blank technical acceptance criteria.

11.3.2 The sample and any required dilution must be analyzed within the contract holding time.

11.3.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.3.4 The Percent Recovery (%R) of each of the DMCs in the sample must be within the recovery limits in Table 10 - Deuterated Monitoring Compound Recovery Limits. Up to three DMCs per sample may fail to meet the recovery limits listed in Table 10 - Deuterated Monitoring Compound Recovery Limits. For TCLP leachate sample analysis, up to two DMCs associated to the TCLP analytes may fail to meet the recovery limits listed in Table 10 – Deuterated Monitoring Compound Recovery Limits.

11.3.5 The EICP area for each of the internal standards in the sample must be within the range of 50%-200% of its response in the most recent opening CCV standard analysis.

11.3.6 The RT shift for each of the internal standards in the sample must be within ±10 seconds of its RT in the most recent opening CCV standard analysis.

11.3.7 Excluding those ions in the solvent front, no ion may saturate the detector. No target analyte concentration may exceed the upper limit of the initial calibration range, unless a more diluted aliquot of the sample is also analyzed according to the procedures in Section 10.2.4.

11.3.8 The Contractor must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted. After a sample that contains a target analyte at a level exceeding the initial calibration range, the Contractor must either:

- Analyze an instrument blank immediately after the contaminated sample. If an autosampler is used, an instrument blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The instrument blanks must meet the technical acceptance criteria for blank analysis (Section 12.1.3.5); or
- Monitor the sample analyzed immediately after the contaminated sample for all analytes that were in the contaminated sample and that exceeded the calibration range. The maximum carryover criteria are as follows: the sample must not contain a concentration above the adjusted CRQL for the target analytes

that exceeded the limits in the contaminated sample. If an auto sampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample must also meet the maximum contamination criteria.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or any samples not meeting the sample technical acceptance criteria will require reanalysis at no additional cost to the EPA.

11.4.2 Corrective actions for failure to meet technical acceptance criteria for instrument performance checks, initial calibration, ICV, CCV, and method blanks must be completed before the analysis of samples.

11.4.3 If the technical acceptance criteria for any of the internal standards and DMCs are not met, check calculations, internal standard and DMC spiking solutions, and instrument performance. It may be necessary to bake out the system to remove the water from the P/T transfer lines, to recalibrate the instrument, or take other corrective action procedures to meet the technical acceptance criteria.

11.4.4 After completing the corrective actions outlined above, the Contractor shall proceed to reanalyzing the sample as appropriate.

11.4.4.1 If the DMC recoveries do not meet the acceptance criteria in the initial sample analysis, reanalyze the sample.

- If the DMC recoveries fail to meet the acceptance criteria in the reanalyzed sample, then submit the data from both analyses. Distinguish between the initial analysis and the reanalysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.2 If the internal standard compound responses do not meet the acceptance criteria in the initial sample analysis, reanalyze the sample.

- If the internal standard compound responses are still noncompliant after the reanalysis, the Contractor shall dilute the original sample by a factor of 2-10 and reanalyze the sample. If the internal standard compound responses are acceptable in any of the subsequent diluted analyses, submit the data from both the reanalysis and the compliant diluted analysis.

- If the internal standard compound responses fail to meet the acceptance criteria in the reanalysis and the subsequent diluted analysis, submit the data from both analysis. Distinguish between the initial analysis, reanalysis, and the diluted analysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.3 If the DMC recoveries in Section 11.4.4.1, the internal standard compound responses in Section 11.4.4.2, or both the DMC recoveries and the internal standard compound responses meet the acceptance criteria in the reanalyzed sample, it indicates that the problem was within the Contractor’s control. Therefore, only submit the data from the reanalysis.

11.4.4.4 If the DMC recoveries or internal standard compound responses in a sample used for the MS/MSD analyses are outside the acceptance criteria, the Contractor shall proceed to the following corrective actions:

- If the DMC recoveries in a sample used for the MS/MSD analyses are outside the acceptance criteria, then the sample shall be reanalyzed only if the DMC recoveries meet the acceptance criteria in both the MS and MSD analyses.
- If the internal standard compound responses do not meet the acceptance criteria, the Contractor shall proceed to the reanalysis in Sections 11.4.4.2 and 11.4.4.3 even if the internal standard compound responses meet the technical acceptance criteria in the MS/MSD analyses.

11.4.5 If the Contractor needs to analyze more than one sample dilution other than the original analysis to have all concentrations of the target analytes within the initial calibration range, contact SMO. SMO will contact the EPA Region for instruction.

11.4.6 All samples to be reported to the EPA must meet the maximum carryover criteria in Section 11.3.8. If any sample fails to meet these criteria, each subsequent analysis must be checked for cross-contamination. The analytical system is considered contaminated until a sample has been analyzed that meets the maximum carryover criteria or an instrument blank has been analyzed that meets the technical acceptance criteria for blanks. If an instrument blank is not analyzed between consecutive samples that have the same analyte with a concentration exceeding the calibration range, then the second sample must be appropriately diluted as indicated in Section 10.2.4 and analyzed. If this analyte in the diluted analysis is detected at or below the adjusted CRQL, then all samples analyzed after the second sample that fail to meet maximum carryover criteria must be reanalyzed. If this analyte in the diluted analysis is detected within the calibration range, then no further corrective action is required.

11.4.7 Corrective Action for Internal Standard Compound Retention Times Outside Acceptance Criteria

11.4.7.1 If the internal standard compound RTs are not within their acceptance criteria, check the instrument for malfunctions. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the samples.

11.4.7.2 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Reanalyze the sample. EXCEPTION: If the internal standard compound RTs in a sample used for an MS or MSD were outside the acceptance criteria, then it should be reanalyzed only if the internal standard RTs were within the acceptance criteria in both the MS/MSD analyses.
- If the internal standard compound RTs are within the acceptance criteria, then the problem was within the Contractor’s control. Therefore, submit only data from the reanalysis when the internal standard compound RTs are within the acceptance limits.

- If the internal standard compound RTs are outside the acceptance criteria in the reanalysis, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables, using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.8 If the required corrective actions for sample reanalysis and/or dilution cannot be performed due to insufficient sample volume, the Contractor shall contact SMO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Summary

There are three different types of blanks required by this method: the method blank, the instrument blank, and the storage blank.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is a volume of a clean reference matrix (reagent water for water samples or a purified solid matrix for soil/sediment samples) spiked with internal standard spiking solution (Section 7.2.2.6) and DMC solution (Section 7.2.2.4), and carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of the method blank is to determine the levels of contamination associated with processing and analysis of samples.

NOTE 1: For soil/sediment samples, if any samples are prepared without the sodium bisulfate preservative, a method blank shall be prepared in the same manner and analyzed in the same 12-hour sequence as the unpreserved samples.

NOTE 2: A leachate blank carried through the TCLP process shall be analyzed with all associated samples.

12.1.2.2 Frequency of Method Blank

12.1.2.2.1 The method blank must be analyzed at least once during every 12-hour period on each GC/MS system used for volatile analysis (see Section 9.2.2 for the definition of the 12-hour period).

12.1.2.2.2 The method blank must be analyzed after the initial calibration sequence (Section 9.3.1) if samples are analyzed before the 12-hour period expires. The method blank must be analyzed after the opening CCV and before any samples, including MS/MSDs or dilutions, are analyzed. A method blank must be analyzed in each 12-hour period in which samples, including dilutions, MS/MSDs, and storage blanks from an SDG are analyzed.

12.1.2.3 Procedure for Method Blank

12.1.2.3.1 For water samples, method blanks shall be analyzed in the same manner as the associated samples, following the procedure described in Section 10.2.1.

12.1.2.3.2 For low-level soil samples, method blanks shall be analyzed in the same manner as the associated samples, following the procedure described in Section 10.2.2.

12.1.2.3.3 For medium-level soil samples, method blanks shall be analyzed in the same manner as the associated samples, following the procedure described in Section 10.2.3.

12.1.2.3.4 For TCLP leachates, the leachate blank shall be analyzed in the same manner as the associated leachate samples, following the procedure described in Section 10.2.1.

12.1.2.3.5 Under no circumstances should method blanks be analyzed at a dilution.

12.1.2.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.0.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.2.2.

12.1.2.5.2 The %R of each of the DMCs in a blank must be within the acceptance windows in Table 10 – Deuterated Monitoring Compound Recovery Limits.

12.1.2.5.3 The blank must meet the sample acceptance criteria listed in Sections 11.3.4 - 11.3.7.

12.1.2.5.4 The concentration of each target analyte found in the method blank must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, except for methylene chloride, acetone, and 2-butanone, which must be less than 2 times the respective CRQL.

12.1.2.5.5 The concentration of each TIC found in the method blank must be less than the CRQL.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.2.6.2 If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further analysis proceeds. It is the Contractor’s responsibility to ensure that method interferences caused by the contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in GCs be eliminated.

12.1.2.6.3 Any method blank that fails to meet the technical acceptance criteria must be reanalyzed. Further, all samples processed within the 12-hour period with a method blank that does not meet the blank technical acceptance criteria will require reanalysis at no additional cost to the EPA.

12.1.3 Instrument Blank

12.1.3.1 Summary of Instrument Blank

An instrument blank is a 5.0 mL aliquot of reagent water spiked with internal standard spiking solution (Section 7.2.2.6) and DMC solution (Section 7.2.2.4), and carried through the entire analytical procedure. Instrument blanks are analyzed after a

sample/dilution that contains a target analyte exceeding the calibration range. The results from the instrument blank analysis indicate whether there is contamination from a previous sample.

12.1.3.2 Frequency of Instrument Blank

Samples may contain target analytes at levels exceeding the calibration. An instrument blank must be analyzed after the sample that exceeds the calibration range (also in the same purge inlet if an autosampler is used) or a sample that exceeds the maximum contamination criteria in Section 11.3.8 must be analyzed. If the instrument blank or sample does not meet the criteria (i.e., contaminated), the system must be decontaminated until an instrument blank meets the blank technical acceptance criteria or a sample meets the maximum carryover criteria.

NOTE: Only the instrument blank that demonstrates that there was no carryover from the previous sample or the instrument blank that demonstrates that the system is clean (Section 12.1.2.5.3) must be reported. Instrument blanks analyzed during the instrument decontamination process that exceed the requirements listed in Section 11.3.8 do not need to be reported.

12.1.3.3 Procedure for Instrument Blank

12.1.3.3.1 Instrument blanks shall be analyzed in the same manner as the associated samples following the procedures outlined in Section 10.0, and in accordance with the protocol of Section 11.3.8.

12.1.3.3.2 Under no circumstances should instrument blanks be analyzed at a dilution.

12.1.3.4 Calculations for Instrument Blank

Perform data analysis and calculations according to Section 11.0.

12.1.3.5 Technical Acceptance Criteria for Instrument Blank

12.1.3.5.1 All instrument blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.3.2.

12.1.3.5.2 The RT shift for each of the internal standards in a blank must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.1.3.5.3 The concentration of each target analyte in the instrument blank must be less than its CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.

12.1.3.5.4 It is the Contractor’s responsibility to ensure that method interferences caused by the contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms, be eliminated.

12.1.3.6 Corrective Action for Instrument Blank

12.1.3.6.1 If a Contractor's instrument blanks exceed the criteria in Section 12.1.3.5, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further analysis proceeds.

12.1.3.6.2 Any instrument blank that fails to meet any technical acceptance criteria described in Section 12.1.3.5 requires reanalysis of the samples analyzed immediately after the instrument blank having any target analytes detected at levels above the CRQLs at no additional cost to the EPA.

12.1.4 Storage Blank

12.1.4.1 Summary of Storage Blank

A storage blank is a volume of a clean reference matrix (reagent water for water samples and preserved soil samples stored at ≤6°, or inert sand for unpreserved soil samples stored at <-7°C). The storage blanks are stored with the samples in the SDG under the same conditions. The storage blank indicates whether contamination may have occurred during storage of samples.

12.1.4.2 Frequency of Storage Blank

A minimum of one storage blank must be analyzed per matrix type (1 for soils and 1 for water samples) and SDG after all samples for the SDG stored in the same manner have been analyzed, unless the SDG contains only ampulated PE samples. Analysis of a storage blank is not required for SDGs that contain only ampulated PE samples.

12.1.4.3 Procedure for Storage Blank

12.1.4.3.1 Upon receipt of the first samples in an SDG, two vials with a clean reference matrix are stored with the samples in the SDG under the same conditions.

NOTE: If the SDG contains samples stored at ≤6°, but not frozen, and samples stored at <-7°C, two storage blanks will be prepared, one for each condition.

12.1.4.3.2 Storage blanks shall be analyzed in the same manner as the associated samples following the procedures outlined in Section 10.0.

12.1.4.3.3 Under no circumstances should storage blanks be analyzed at a dilution.

12.1.4.4 Calculations for Storage Blank

Perform data analysis and calculations according to Section 11.0.

12.1.4.5 Technical Acceptance Criteria for Storage Blank

12.1.4.5.1 All storage blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.4.2.

12.1.4.5.2 The storage blank must be analyzed on a GC/MS system that also meets the technical acceptance criteria for the method blank.

12.1.4.5.3 The %R of each of the DMCs in the blank must be within the acceptance windows in Table 10 – Deuterated Monitoring Compound Recovery Limits.

12.1.4.5.4 The EICP area for each of the internal standards in a blank must be within the range of 50%-200% of its response in the most recent opening CCV standard analysis.

12.1.4.5.5 The RT shift for each of the internal standards in a blank must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.1.4.5.6 The concentration of each target analyte found in the storage blank must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, except for methylene chloride, acetone, and 2-butanone, which must be less than 2 times the respective CRQL.

12.1.4.5.7 It is the Contractor's responsibility to ensure that method interferences caused by the contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.4.6 Corrective Action for Storage Blank

12.1.4.6.1 If a Contractor's storage blanks exceed the criteria in Section 12.1.4.5, the Contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further analysis proceeds.

12.1.4.6.2 If the storage blank does not meet the technical acceptance criteria for blank analyses in Section 12.1.4.5, correct system problems and reanalyze the storage blank.

12.1.4.6.3 If, upon reanalysis, the storage blank meets the criteria, the problem occurred during the analysis and the reanalyzed storage blank results must be reported. If upon reanalysis, the storage blank still does not meet the criteria, the problem occurred during storage. The Laboratory Manager or their designee must address the problem in the SDG Narrative and discuss the corrective actions implemented to prevent future occurrences.

NOTE: A copy of the storage blank data must also be retained by the Contractor and be made available for inspection during on-site laboratory evaluations.

12.2 Matrix Spike and Matrix Spike Duplicate

12.2.1 Summary of Matrix Spike and Matrix Spike Duplicate

To evaluate the effects of the sample matrix on the methods used for volatile analyses, the EPA has prescribed a mixture of volatile target analytes to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method. An MS/MSD shall only be analyzed if requested by the EPA Region (through SMO) or specified on the TR/COC Record.

12.2.2 Frequency of Matrix Spike and Matrix Spike Duplicate

12.2.2.1 If requested, an MS/MSD must be performed for each group of 20 field samples of a similar matrix in an SDG. An MS/MSD should be analyzed for each sample matrix (water/soil) and each level (low/med).

12.2.2.2 The Contractor shall not perform MS/MSD analysis on any of the field QC or PE samples.

12.2.2.3 If an insufficient number of sample vials were received to perform an MS/MSD, and MS/MSD are required, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the EPA Region for instructions. The EPA Region has the option to cancel the MS/MSD analysis. SMO will notify the Contractor of the resolution. The Contractor shall document the decision in the SDG Narrative.

12.2.2.4 If it appears that the EPA Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2.1, the Contractor shall contact SMO. SMO will contact the EPA Region to determine which samples should have an MS/MSD analysis performed on them. SMO will notify the Contractor of the EPA Region's decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 When a Contractor receives only PE sample(s), no MS/MSD shall be performed within that SDG.

12.2.3 Procedure for Preparing Matrix Spike and Matrix Spike Duplicate

12.2.3.1 To prepare an MS/MSD for water samples, add 20 µL of the matrix spiking solution (Section 7.2.2.5) to each of the 5.0 mL aliquots of the sample chosen for spiking. Process the samples according to Section 10.2.1. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each Matrix Spike analyte.

12.2.3.2 To prepare an MS/MSD for low-level soil/sediment samples, add 20 µL of the matrix spiking solution (Section 7.2.2.5) either manually by puncturing the septum with a small-gauge needle or automatically by the P/T system just prior to analysis. Analyze the MS/MSD samples by the procedure described in Section 10.2.2. Do not further dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range.

12.2.3.3 To prepare an MS/MSD for medium-level soil/sediment samples, add 4.0 mL of methanol and 1.0 mL of the matrix spiking solution (Section 7.2.2.5) to each of the two aliquots of the soil/sediment sample chosen for spiking. Analyze the MS/MSD sample according to Section 10.2.3.

12.2.3.3.1 In the cases where methanol has been added as a preservative, do not add additional methanol. Add only 1.0 mL of the matrix spiking solution to each of the two aliquots of the soil/sediment sample chosen for spiking.

12.2.3.3.2 Process samples according to Section 10.2.3. This results in a 2,500 µg/kg concentration of each Matrix Spike analyte when added to a 5 g sample. Add a 100 µL aliquot of this extract to 4.9 mL of water for purging (per Sections 10.2.3.5 and 10.2.3.6).

12.2.3.4 MS/MSD samples shall be analyzed at the same dilution as the least diluted aliquot for which the sample results will be reported to the EPA. Sample dilutions must be performed in accordance with Section 10.2.4. Do not further dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range.

12.2.4 Calculations for Matrix Spike and Matrix Spike Duplicate

12.2.4.1 Calculate the concentrations of the Matrix Spike analytes using the same equations as used for target analytes (Equations 7, 8, and 9). Calculate the recovery of each Matrix Spike analyte using the following equation:

EQ. 14 Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each analyte in the MS/MSD using the following equation:

EQ. 15 Relative Percent Difference

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

WHERE,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

NOTE: The vertical bars in the equation above indicate the absolute value of the difference.

12.2.5 Technical Acceptance Criteria for Matrix Spike and Matrix Spike Duplicate

12.2.5.1 All MS/MSDs must be analyzed on a GC/MS system meeting the BFB, initial calibration, ICV, CCV, and blank technical acceptance criteria, and at the frequency described in Section 12.2.2.

12.2.5.2 The MS/MSD must be analyzed within the contract holding time.

12.2.5.3 The RT shift for each of the internal standards in the MS/MSD must be within 10 seconds of its RT in the most recent opening CCV standard analysis.

12.2.5.4 The limits for MS analyte recovery and RPD are given in Table 11 – Matrix Spike Recovery and Relative Percent Difference Limits. As these limits are only advisory, no further action by the Contractor is required.

12.2.6 Corrective Action for Matrix Spike and Matrix Spike Duplicate

Any MS/MSD that does not meet the technical acceptance criteria in Sections 12.2.5.1 and 12.2.5.3 must be reanalyzed at no additional cost to the EPA.

12.3 Laboratory Control Sample

Not applicable to this method.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed, the Method Detection Limit (MDL) for each volatile target analyte shall be determined on each instrument used for analysis. MDL determination is matrix-specific and level-specific (i.e., the MDL shall be determined for water, low-level soil/sediment, and medium-level soil/sediment samples). The MDLs must be determined annually thereafter or after major instrument maintenance. Major instrument maintenance includes, but is not limited to: cleaning or replacement of the mass spectrometer source, mass filters (e.g., quadrupole, ion trap, etc.), or electron multiplier (or similar device); and replacement or overhaul of the P/T device. A new MDL study will not be required after changing the GC column, as long as the replacement has the same length, inner diameter, and stationary phase.

12.4.2 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.3 The determined concentration of the MDL must be less than the CRQL listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 - Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and submitted to the EPA within seven (7) days of study completion. This schedule and the designated recipients are specified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 of Exhibit D – Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 of Exhibit D – Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, July 2002.

16.2 U.S. Environmental Protection Agency, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Method 524.2, Revision 4, August 1992.

16.3 U.S. Environmental Protection Agency, Purge-and-Trap for Aqueous Samples, Method 5030C, Revision 3, May 2003.

16.4 U.S. Environmental Protection Agency, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C, Revision 3, August 2006.

16.5 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE REGISTRY NUMBERS

Systematic Name	EPA Registry Name	Synonym	CAS #
Methane, dichlorodifluoro-	CFC-12	Dichlorodifluoromethane	75-71-8
Methane, chloro-	Chloromethane	Methyl chloride	74-87-3
Ethene, chloro-	Vinyl Chloride	Vinyl chloride	75-01-4
Methane, bromo-	Methyl Bromide	Methyl bromide	74-83-9
Ethane, chloro-	Chloroethane	Ethyl chloride	75-00-3
Methane, trichlorofluoro-	CFC-11	Fluorotrichloromethane	75-69-4
Ethene, 1,1-dichloro-	1,1-Dichloroethylene	Vinylidene chloride	75-35-4
Ethane, 1,1,2-trichloro-1,2,2-trifluoro-	CFC-113	Freon 113	76-13-1
2-Propanone	Acetone	Dimethyl ketone	67-64-1
Carbon disulfide	Carbon disulfide	Dithiocarbonic anhydride	75-15-0
Acetic acid, methyl ester	Methyl acetate	Methyl acetate	79-20-9
Methane, dichloro	Methylene chloride	Dichloromethane	75-09-2
Ethene, 1,2-dichloro-, (1E)-	trans-1,2-Dichloroethylene	Ethylene, 1,2-dichloro-, (E)-	156-60-5
Propane, 2-methoxy-2-methyl-	Methyl tert-butyl ether	t-Butyl methyl ether	1634-04-4
Ethane, 1,1-dichloro-	1,1-Dichloroethane	Ethylidene dichloride	75-34-3
Ethene, 1,2-dichloro-, (1Z)-	cis-1,2-Dichloroethylene	Ethylene, 1,2-dichloro-, (Z)-	156-59-2
2-Butanone	Methyl ethyl ketone	Butan-2-one	78-93-3
Methane, bromochloro-	Halon 1011	Chlorobromomethane	74-97-5
Methane, trichloro-	Chloroform	Trichloromethane	67-66-3
Ethane, 1,1,1-trichloro-	1,1,1-Trichloroethane	1,1,1-TCE	71-55-6
Cyclohexane	Cyclohexane	Hexahydrobenzene	110-82-7
Methane, tetrachloro-	Carbon tetrachloride	Tetrachlorocarbon	56-23-5
Benzene	Benzene	Benzol	71-43-2
Ethane, 1,2-dichloro-	1,2-Dichloroethane	Ethylene dichloride	107-06-2
Ethene, 1,1,2-trichloro-	Trichloroethylene	Ethylene, trichloro-	79-01-6
Cyclohexane, methyl-	Methylcyclohexane	Hexahydrotoluene	108-87-2
Propane, 1,2-dichloro-	1,2-Dichloropropane	Propylene dichloride	78-87-5
Methane, bromodichloro-	Dichlorobromomethane	Bromodichloromethane	75-27-4
1-Propene, 1,3-dichloro-, (Z)-	cis-1,3-Dichloropropene	cis-1,3-Dichloropropylene	10061-01-5

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
2-Pentanone, 4-methyl-	Methyl isobutyl ketone	2-Methylpropyl methyl ketone	108-10-1
Benzene, methyl-	Toluene	Methylbenzol	108-88-3
1-Propene, 1,3-dichloro-, (1E)-	trans-1,3-Dichloropropene	trans-1,3-Dichloropropylene	10061-02-6
Ethane, 1,1,2-trichloro-	1,1,2-Trichloroethane	1,1,2-TCA	79-00-5
Ethene, 1,1,2,2-tetrachloro-	Tetrachloroethylene	Tetrachlorethene	127-18-4
2-Hexanone	2-Hexanone	Methyl n-butyl ketone	591-78-6
Methane, dibromochloro-	Chlorodibromomethane	Dibromochloromethane	124-48-1
Ethane, 1,2-dibromo-	Ethylene Dibromide	1,2-Dibromoethane	106-93-4
Benzene, chloro-	Chlorobenzene	Phenyl chloride	108-90-7
Benzene, ethyl-	Ethylbenzene	Phenylethane	100-41-4
Benzene, 1,2-dimethyl-	o-Xylene	1,2-Dimethylbenzene	95-47-6
Benzene, (1,3 and 1,4)-dimethyl-	m,p-Xylene	(1,3 and 1,4)-Dimethyl benzene	179601-23-1
Benzene, ethenyl-	Styrene	Vinyl Benzene	100-42-5
Methane, tribromo-	Tribromomethane	Bromoform	75-25-2
Benzene, (1-methylethyl)-	Cumene	Isopropylbenzene	98-82-8
Ethane, 1,1,2,2-tetrachloro-	1,1,2,2-Tetrachloroethane	Acetylene tetrachloride	79-34-5
Benzene, 1,3-dichloro-	m-Dichlorobenzene	m-Phenylene dichloride	541-73-1
Benzene, 1,4-dichloro-	p-Dichlorobenzene	p-Chlorophenyl chloride	106-46-7
Benzene, 1,2-dichloro-	o-Dichlorobenzene	ortho-Dichlorobenzene	95-50-1
Propane, 1,2-dibromo-3-chloro-	1,2-Dibromo-3-chloropropane	Dibromochloropropane	96-12-8
Benzene, 1,2,4-trichloro-	1,2,4-Trichlorobenzene	1,2,4-Trichlorobenzol	120-82-1
Benzene, 1,2,3-trichloro-	1,2,3-Trichlorobenzene	Vic-Trichlorobenzene	87-61-6
Internal Standards			
Benzene-d5, chloro-	Chlorobenzene-d5	Chlorobenzene-d5	3114-55-4
Benzene, 1,4-difluoro	1,4-Difluorobenzene	p-Difluorobenzene	540-36-3
Benzene-1,2,4,5-d4, 3,6-dichloro	1,4-Dichlorobenzene-d4	1,4-Dichloro-2,3,5,6-tetradeuterobenzene	3855-82-1

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
DMCs			
Ethene-d3, chloro-	Vinyl chloride-d3	Vinyl chloride-d3	6745-35-3
Ethane-d5, chloro-	Chloroethane-d5	Chloroethane-d5	19199-91-8
Ethene-1,1-d2, dichloro-	1,1-Dichloroethene-d2	1,1-Dichloroethene-d2	22280-73-5
2-Butanone-1,1,1,3,3-d5	2-Butanone-d5	2-Butanone-d5	24313-50-6
Methane-d, trichloro-	Chloroform-d	Chloroform-d	865-49-6
Ethane-1,1,2,2-d4, 1,2-dichloro-	1,2-Dichloroethane-d4	1,2-Dichloroethane-d4	17060-07-0
Benzene-1,2,3,4,5,6-d6	Benzene-d6	Benzene-d6	1076-43-3
Propane-1,1,1,2,3,3-d6, 2,3-dichloro-	1,2-Dichloropropane-d6	1,2-Dichloropropane-d6	93952-08-0
Benzene-d5, methyl-d3-	Toluene-d8	Perdeuterotoluene	2037-26-5
1-Propene-1,2,3,3-d4, 1,3-dichloro-(E)-	Trans-1,3-Dichloropropene-d4	Trans-1,3-Dichloropropene-d4	93951-86-1
2-Hexanone-1,1,1,3,3-d5		2-Hexanone-d5	4840-82-8
Ethane-1,2-d2, 1,1,2,2-tetrachloro-	1,1,2,2-Tetrachloroethane-d2	1,1,2,2-Tetrachloroethane-d2	33685-54-0
Benzene-1,2,3,4-d4, 5,6-dichloro-	1,2-Dichlorobenzene-d4	1,2-Dichloro-3,4,5,6-tetradeuterobenzene	2199-69-1

TABLE 2. 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 80.0% of mass 95
95	base peak, 100% Relative Abundance
96	5.0 - 9.0% of mass 95 (see NOTE)
173	less than 2.0% of mass 174
174	50.0 - 120% of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101% of mass 174
177	5.0 - 9.0% of mass 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

TABLE 3. VOLATILE DEUTERATED MONITORING COMPOUNDS
AND THE ASSOCIATED TARGET ANALYTES

Vinyl chloride-d_3 (DMC-1)	**Chloroethane-d_5 (DMC-2)**	**1,1-Dichloroethene-d_2 (DMC-3)**
Vinyl chloride	Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon disulfide	trans-1,2-Dichloroethene cis-1,2-Dichloroethene 1,1-Dichloroethene
2-Butanone-d_5 (DMC-4)	**Chloroform-d (DMC-5)**	**1,2-Dichloroethane-d_4 (DMC-6)**
Acetone 2-Butanone	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
Benzene-d_6 (DMC-7)	**1,2-Dichloropropane-d_6 (DMC-8)**	**Toluene-d_8 (DMC-9)**
Benzene	Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene-d_4 (DMC-10)	**2-Hexanone-d_5 (DMC-11)**	**1,1,2,2-Tetrachloroethane-d_2 (DMC-12)**
cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	4-Methyl-2-pentanone 2-Hexanone	1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene-d_4 (DMC-13)		
Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene		

TABLE 4. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR VOLATILE ORGANIC COMPOUNDS

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
Dichlorodifluoromethane	0.010	0.010	25.0	±40.0	±50.0
Chloromethane	0.010	0.010	20.0	±30.0	±50.0
Vinyl chloride	0.010	0.010	20.0	±25.0	±50.0
Bromomethane	0.010	0.010	40.0	±30.0	±50.0
Chloroethane	0.010	0.010	40.0	±25.0	±50.0
Trichlorofluoromethane	0.010	0.010	40.0	±30.0	±50.0
1,1-Dichloroethene	0.060	0.060	20.0	±20.0	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.050	0.050	25.0	±25.0	±50.0
Acetone	0.010	0.010	40.0	±40.0	±50.0
Carbon disulfide	0.100	0.100	20.0	±25.0	±25.0
Methyl acetate	0.010	0.010	40.0	±40.0	±50.0
Methylene chloride	0.010	0.010	40.0	±30.0	±50.0
trans-1,2-Dichloroethene	0.100	0.100	20.0	±20.0	±25.0
Methyl tert-butyl ether	0.100	0.100	40.0	±25.0	±50.0
1,1-Dichloroethane	0.300	0.300	20.0	±20.0	±25.0
cis-1,2-Dichloroethene	0.200	0.200	20.0	±20.0	±25.0
2-Butanone	0.010	0.010	40.0	±40.0	±50.0
Bromochloromethane	0.100	0.100	20.0	±20.0	±25.0
Chloroform	0.300	0.300	20.0	±20.0	±25.0
1,1,1-Trichloroethane	0.050	0.050	20.0	±25.0	±25.0
Cyclohexane	0.010	0.010	40.0	±25.0	±50.0
Carbon tetrachloride	0.100	0.100	20.0	±25.0	±25.0
Benzene	0.200	0.200	20.0	±20.0	±25.0
1,2-Dichloroethane	0.070	0.070	20.0	±20.0	±25.0
Trichloroethene	0.200	0.200	20.0	±20.0	±25.0
Methylcyclohexane	0.050	0.050	40.0	±25.0	±50.0
1,2-Dichloropropane	0.200	0.200	20.0	±20.0	±25.0
Bromodichloromethane	0.300	0.300	20.0	±20.0	±25.0
cis-1,3-Dichloropropene	0.300	0.300	20.0	±20.0	±25.0
4-Methyl-2-pentanone	0.030	0.030	25.0	±30.0	±50.0
Toluene	0.300	0.300	20.0	±20.0	±25.0
trans-1,3-Dichloropropene	0.200	0.200	20.0	±20.0	±25.0
1,1,2-Trichloroethane	0.200	0.200	20.0	±20.0	±25.0
Tetrachloroethene	0.100	0.100	20.0	±20.0	±25.0
2-Hexanone	0.010	0.010	40.0	±40.0	±50.0

TABLE 4. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR VOLATILE ORGANIC COMPOUNDS (CON'T)

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
Dibromochloromethane	0.200	0.200	20.0	±20.0	±25.0
1,2-Dibromoethane	0.200	0.200	20.0	±20.0	±25.0
Chlorobenzene	0.400	0.400	20.0	±20.0	±25.0
Ethylbenzene	0.400	0.400	20.0	±20.0	±25.0
m,p-Xylene	0.200	0.200	20.0	±20.0	±25.0
o-Xylene	0.200	0.200	20.0	±20.0	±25.0
Styrene	0.200	0.200	20.0	±20.0	±25.0
Bromoform	0.100	0.100	20.0	±25.0	±50.0
Isopropylbenzene	0.400	0.400	20.0	±25.0	±25.0
1,1,2,2-Tetrachloroethane	0.200	0.200	20.0	±25.0	±25.0
1,3-Dichlorobenzene	0.500	0.500	20.0	±20.0	±25.0
1,4-Dichlorobenzene	0.600	0.600	20.0	±20.0	±25.0
1,2-Dichlorobenzene	0.600	0.600	20.0	±20.0	±25.0
1,2-Dibromo-3-chloropropane	0.010	0.010	25.0	±30.0	±50.0
1,2,4-Trichlorobenzene	0.400	0.400	20.0	±30.0	±50.0
1,2,3-Trichlorobenzene	0.400	0.400	25.0	±30.0	±50.0
Deuterated Monitoring Compounds					
Vinyl chloride-d_3	0.010	0.010	20.0	±30.0	±50.0
Chloroethane-d_5	0.010	0.010	40.0	±30.0	±50.0
1,1-Dichloroethene-d_2	0.050	0.050	20.0	±25.0	±25.0
2-Butanone-d_5	0.010	0.010	40.0	±40.0	±50.0
Chloroform-d	0.300	0.300	20.0	±20.0	±25.0
1,2-Dichloroethane-d_4	0.060	0.060	20.0	±25.0	±25.0
Benzene-d_6	0.300	0.300	20.0	±20.0	±25.0
1,2-Dichloropropane-d_6	0.200	0.200	20.0	±20.0	±25.0
Toluene-d_8	0.300	0.300	20.0	±20.0	±25.0
trans-1,3-Dichloropropene-d_4	0.200	0.200	20.0	±20.0	±25.0
2-Hexanone-d_5	0.010	0.010	40.0	±40.0	±50.0
1,1,2,2-Tetrachloroethane-d_2	0.200	0.200	20.0	±25.0	±25.0
1,2-Dichlorobenzene-d_4	0.400	0.400	20.0	±20.0	±25.0

[1] If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

TABLE 5. PURGE-AND-TRAP ANALYTICAL CONDITIONS

Purge Conditions	
Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ±0.1 min.
Purge Flow Rate:	25-40 mL/min.
Purge Temperature:	Ambient temperature for water or medium-level soil/sediment samples (required for medium-level soil/sediment samples, suggested for water samples), and 40°C for low-level soil/sediment samples.
Desorb Conditions	
Desorb Temperature:	180°C
Desorb Flow Rate:	15 mL/min.
Desorb Time:	4.0 ±0.1 min.
Trap Reconditioning Conditions	
Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 ±0.1 min. (minimum). A longer time may be required to bake contamination or water from the system.

NOTE: Higher purge temperatures may be used provided that manufacturer's instructions are followed and technical acceptance criteria are met for all standards, samples, and blanks. Certain target analytes, such as methyl tert-butyl ether (MTBE), may decompose at high purge temperatures in samples that have been acid preserved.

TABLE 6. GAS CHROMATOGRAPH ANALYTICAL CONDITIONS

Capillary Columns	
Carrier Gas:	Helium
Flow Rate:	15 mL/min.
Initial Temperature:	10°C
Initial Hold Time:	1.0–5.0 (±0.1) min.
Ramp Rate:	6°C/min.
Final Temperature:	160°C
Final Hold Time:	Until 3 min. after all analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 2 – Low/Medium Volatiles Target Analyte List and Contract Required Quantitation Limits, elute (required)

TABLE 7. MASS SPECTROMETER ANALYTICAL CONDITIONS

Electron Energy	70 volts (nominal)
Mass Range	35-300 u
Ionization Mode	Electron ionization (EI)
Scan Time	To give at least 5 scans per peak, not to exceed 2 sec. per scan.

TABLE 8. CHARACTERISTIC IONS FOR VOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,63
1,1,2-Trichloro-1,2,2-trifluoroethane	101	85,151
Acetone	43	58
Carbon disulfide	76	78
Methyl acetate	43	74
Methylene chloride	84	49,86
trans-1,2-Dichloroethene	96	61,98
Methyl tert-butyl ether	73	43,57
1,1-Dichloroethane	63	65,83
cis-1,2-Dichloroethene	96	61,98
2-Butanone	43*	72
Chloroform	83	85
Bromochloromethane	128	49,130,51
1,1,1-Trichloroethane	97	99,61
Cyclohexane	56	69,84
Carbon tetrachloride	117	119
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	95	97,132,130
Methylcyclohexane	83	55,98
1,2-Dichloropropane	63	112
Bromodichloromethane	83	85,127
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58,100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	83,85,99,132,134
Tetrachloroethene	164	129,131,166
2-Hexanone	43	58,57,100
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Chlorobenzene	112	77,114
Ethylbenzene	91	106
m,p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78

*m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TABLE 8. CHARACTERISTIC IONS FOR VOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS (CON'T)

Analyte	**Primary Quantitation Ion**	**Secondary Ion(s)**
Bromoform	173	175,254
Isopropylbenzene	105	120,77
1,1,2,2-Tetrachloroethane	83	85,131
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene	146	111,148
1,2-Dibromo-3-chloropropane	75	157,155
1,2,4-Trichlorobenzene	180	182,145
1,2,3-Trichlorobenzene	180	182,145
Deuterated Monitoring Compounds		
Vinyl chloride-d_3	65	67
Chloroethane-d_5	69	71,51
1,1-Dichloroethene-d_2	63	98,65
2-Butanone-d_5	46	77
Chloroform-d	84	86,47,49
1,2-Dichloroethane-d_4	65	67,51
Benzene-d_6	84	82,54,52
1,2-Dichloropropane-d_6	67	65,46,42
Toluene-d_8	98	100,42
trans-1,3-Dichloropropene-d_4	79	81,42
2-Hexanone-d_5	63	46
1,1,2,2-Tetrachloroethane-d_2	84	86
1,2-Dichlorobenzene-d_4	152	150
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115,150
1,4-Difluorobenzene	114	63,88
Chlorobenzene-d_5	117	82,119

TABLE 9. VOLATILE TARGET ANALYTES AND DEUTERATED MONITORING COMPOUNDS WITH ASSOCIATED INTERNAL STANDARDS FOR QUANTITATION

1,4-Difluorobenzene (IS)	**Chlorobenzene-d_5 (IS)**	**1,4-Dichlorobenzene-d_4 (IS)**
Dichlorodifluoromethane	1,1,1-Trichloroethane	Bromoform
Chloromethane	Cyclohexane	1,3-Dichlorobenzene
Vinyl chloride	Carbon tetrachloride	1,4-Dichlorobenzene
Bromomethane	Benzene	1,2-Dichlorobenzene
Chloroethane	Trichloroethene	1,2-Dibromo-3-chloropropane
Trichlorofluoromethane	Methylcyclohexane	
1,1-Dichloroethene	1,2-Dichloropropane	1,2,4-Trichlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Bromodichloromethane	1,2,3-Trichlorobenzene
Acetone	cis-1,3-Dichloropropene	1,2-Dichlorobenzene-d_4 (DMC)
Carbon disulfide	4-Methyl-2-pentanone	
Methyl acetate	Toluene	
Bromochloromethane	trans-1,3-Dichloropropene	
Methylene chloride	1,1,2-Trichloroethane	
trans-1,2-Dichloroethene	Tetrachloroethene	
Methyl tert-butyl ether	2-Hexanone	
1,1-Dichloroethane	Dibromochloromethane	
cis-1,2-Dichloroethene	1,2-Dibromoethane	
2-Butanone	Chlorobenzene	
Chloroform	Ethylbenzene	
1,2-Dichloroethane	m,p-Xylene	
Vinyl chloride-d_3 (DMC)	o-Xylene	
Chloroethane-d_5 (DMC)	Styrene	
1,1-Dichloroethene-d_2 (DMC)	Isopropylbenzene	
2-Butanone-d_5 (DMC)	1,1,2,2-Tetrachloroethane	
Chloroform-d (DMC)	Benzene-d_6 (DMC)	
1,2-Dichloroethane-d_4 (DMC)	1,2-Dichloropropane-d_6 (DMC)	
	trans-1,3-Dichloropropene-d_4 (DMC)	
	Toluene-d_8 (DMC)	
	2-Hexanone-d_5 (DMC)	
	1,1,2,2-Tetrachloroethane-d_2 (DMC)	

TABLE 10. DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

Compound	Percent Recovery for Water Samples	Percent Recovery for Soil Samples
Vinyl chloride-d_3	60-135	30-150
Chloroethane-d_5	70-130	30-150
1,1-Dichloroethene-d_2	60-125	45-110
2-Butanone-d_5	40-130	20-135
Chloroform-d	70-125	40-150
1,2-Dichloroethane-d_4	70-125	70-130
Benzene-d_6	70-125	20-135
1,2-Dichloropropane-d_6	70-120	70-120
Toluene-d_8	80-120	30-130
trans-1,3-Dichloropropene-d_4	60-125	30-135
2-Hexanone-d_5	45-130	20-135
1,1,2,2-Tetrachloroethane-d_2	65-120	45-120
1,2-Dichlorobenzene-d_4	80-120	75-120

NOTE: The recovery limits for any of the compounds listed above may be expanded at any time during the period of performance if the EPA determines that the limits are too restrictive.

TABLE 11. MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Percent Recovery Water	RPD Water	Percent Recovery Soil	RPD Soil
1,1-Dichloroethene	61-145	0-14	59-172	0-22
Trichloroethene	71-120	0-14	62-137	0-24
Benzene	76-127	0-11	66-142	0-21
Toluene	76-125	0-13	59-139	0-21
Chlorobenzene	75-130	0-13	60-133	0-21

EXHIBIT D

SEMIVOLATILE ORGANIC COMPOUNDS ANALYSIS

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Exhibit D – Semivolatile Organic Compounds Analysis

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water, leachate derived from the Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP), and soil/sediment samples from hazardous waste sites to determine the presence and concentration of the semivolatile organic analytes (SVOA) contained in the Target Analyte List (TAL) for semivolatiles in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. The method, based on the U.S. Environmental Protection Agency (EPA) SW-846 Method 8270D, covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to Gas Chromatography (GC). The method involves solvent extraction of the matrix sample, characterization to determine the appropriate analytical protocol to be used, followed by appropriate cleanup procedure and GC/Mass Spectrometry (MS) analysis to determine the SVOAs present in the sample.

1.2 If requested, sample extracts will be analyzed for the specific group of Polynuclear Aromatic Hydrocarbon (PAH) analytes and pentachlorophenol (PCP) by GC/MS, using the full scan method and/or the Selected Ion Monitoring (SIM) technique. If a SIM analysis is requested, a full scan analysis using the low-level method must be performed first. If all PAHs and PCP are detected at or above the Contract Required Quantitation Limits (CRQLs) during the full scan analysis using the low-level method, then a SIM analysis is not to be performed and this should be documented in the Sample Delivery Group (SDG) Narrative. If only PAHs and PCP are requested by the full scan method, quantitate and report only these target analytes along with the associated Deuterated Monitoring Compounds (DMCs) and internal standards for calibration standards, method blanks, and samples. The SIM analysis is not required for a specific PAH target analyte or PCP which is detected at or above the sample-adjusted CRQL in the full scan analysis. However, if any single PAH analyte or PCP exceeds the calibration range, do not proceed with the SIM method for any of the target analytes scheduled for SIM analysis.

1.3 Problems that have been associated with the following analytes using this method include:

- 3,3'-Dichlorobenzidine and 4-Chloroaniline can be subject to oxidative losses during solvent concentration.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the GC inlet, chemical reactions in acetone solution, and photochemical decomposition.
- N-nitrosodiphenylamine decomposes in the GC inlet forming diphenylamine and consequently, may be detected as diphenylamine.
- PCP, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-nitroaniline are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

2.0 SUMMARY OF METHOD

2.1 Water/TCLP or SPLP Leachate

A 1 Liter (L) aliquot of sample is mixed with DMCs, acidified to pH 2.0, and extracted with methylene chloride using a continuous liquid-liquid extractor. Separatory funnel extraction is NOT permitted. The methylene chloride extract is dried with sodium sulfate (or an equivalent drying agent such as Hydromatrix™), concentrated, and subjected to Gel Permeation Chromatography (GPC) cleanup. GPC is required when higher molecular weight compounds are present that interfere with the analyses of target analytes; GPC is optional for all other circumstances. The extract is then analyzed by GC/MS for extractable organics.

2.2 Soil/Sediment

2.2.1 Low-Level Soil/Sediment

A 30 gram (g) aliquot of soil/sediment is spiked with DMCs, mixed with anhydrous powdered sodium sulfate (or Hydromatrix™) and extracted with 1:1 (v/v) methylene chloride/acetone solution using an ultrasonic probe, a Soxhlet extractor, or a pressurized fluid extractor. The extract is concentrated, subjected to GPC cleanup, and analyzed by GC/MS for extractable organics.

The Contractor must determine whether a soil/sediment sample should be analyzed by the low-level or medium-level method, using an EPA-approved screening procedure or an in-house laboratory screening procedure.

2.2.2 Medium-Level Soil/Sediment

Approximately 1 g aliquot of sample is mixed with anhydrous powdered sodium sulfate (or Hydromatrix™) and DMCs in a vial and extracted with methylene chloride. The methylene chloride extract is subjected to GPC cleanup and optional silica gel cleanup (SW-846 Method 3630C), prior to analysis by GC/MS for extractable organics.

2.3 Wipes

Not applicable to this method.

2.4 Waste

Not applicable to this method.

2.5 Non-Target Compounds

Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library. Non-target compounds are quantitated by comparing the area response from the total Reconstructed Ion Chromatogram (RIC) for the non-target compound peaks to the area response produced by the nearest internal standard. A Relative Response Factor (RRF) of 1 is assumed.

3.0 DEFINITIONS

See Exhibit G – Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts and/or elevated baselines in the Extracted Ion Current Profiles (EICPs). These materials must be routinely demonstrated to be free from interferences under the sample preparation and analysis conditions by analyzing laboratory method blanks.

4.2 Matrix Interferences

Matrix interferences may be caused by compounds that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled.

5.0 SAFETY

See Section 12.0 of Exhibit D – Introduction to Organic Analytical Methods.

5.1 Reagents

Concentrated sulfuric acid presents some hazards and is moderately toxic and extremely irritating to skin and mucous membranes. Use this reagent in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with this reagent.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the SDG Narrative.

6.1 General Laboratory Equipment

6.1.1 Balances

6.1.1.1 Top loading, capable of weighing accurately to ±0.01 g.

6.1.1.2 Analytical, capable of weighing accurately to ±0.0001 g.

6.1.1.3 A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each type of balance and be accurate to ±0.01 g and ±0.0001 g, respectively. The masses that are used to check the balances daily must be checked on a monthly basis using NIST-traceable known reference masses (Class ‘0’ or Class ‘1’) as defined by ASTM E617-97(2008) or equivalent (e.g., earlier Class ‘S’ defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified at least every five years, or sooner if there is reason to believe damage

(corrosion, nicks)has occurred. The Contractor shall maintain documentation that demonstrates these criteria have been met.

6.1.2 Beakers - 100 milliliters (mL), 125 mL, 250 mL, and 400 mL.

6.1.3 Centrifuge, Table top (optional).

6.1.3.1 Centrifuge Tube - 12-15 mL with 19 millimeter (mm) ground-glass joint (optional).

6.1.4 Graduated Cylinder Class A - 1 L and 100 mL capacity.

6.1.5 Desiccator.

6.1.6 Erlenmeyer Flasks - 250 mL.

6.1.7 Volumetric Flask, Class A – 5.0, 10, 20, 50, 100, 250, and 500 mL.

6.1.8 Magnetic Stirring Bar - Polytetrafluoroethylene (PTFE) coated, at least 4 centimeters (cm) long.

6.1.9 Ovens – drying, capable of maintaining 105°C (±5°C).

6.1.10 pH Meter - With a combination glass electrode. Calibrate according to manufacturer's instructions. The pH meter shall be calibrated prior to each use, using reference standards bracketing the range expected in samples. The pH reference standards shall be replaced when their expiration dates have passed.

6.1.11 pH Paper – Wide range.

6.1.12 Pipettes (Calibrated) - Glass volumetric, 1.0 mL or 2.0 mL. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.1.13 Spatula - Stainless steel or PTFE.

6.1.14 Syringes – 10 microliters (µL), 25 µL, 100 µL, and 1000 µL.

6.1.15 Vials and Caps - 10 mL (optional), with screw-cap and PTFE or aluminum foil liner; autosampler vial with 2 mL capacity for GC autosampler.

6.1.16 Weigh Dish – Porcelain crucibles or disposable aluminum weighing pans.

6.2 Glassware/Extraction/Cleanup Equipment

6.2.1 Automated Soxhlet Extraction System - With temperature-controlled oil bath. Silicone oil must not be used because it destroys the rubber parts. The apparatus must be used in a hood.

6.2.1.1 Cellulose or Glass Extraction Thimble, 26 mm ID x 60 mm.

6.2.1.2 Glass Extraction Cups.

6.2.1.3 Thimble Adapters.

6.2.1.4 Viton Seals.

6.2.2 Soxhlet Extraction, Manual

6.2.2.1 Allihn Condenser.

6.2.2.2 Soxhlet Extractor body, 40 mm ID.

6.2.2.3 Round bottom flask, 500 mL.

6.2.3 Borosilicate Glass Wool - Rinsed with methylene chloride.

6.2.4 Continuous Liquid-Liquid Extractors - Equipped with PTFE or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf extractor) or hydrophobic membrane-based extractor.

6.2.5 Drying Column - 400 mm x 19 mm ID chromatographic column with coarse frit (substitution of a small pad of borosilicate glass wool for the frit will help prevent cross-contamination of sample extracts).

6.2.6 Gel Permeation Chromatography Cleanup System

6.2.6.1 GPC System - Systems that perform satisfactorily have been assembled from the following components: a High Performance Liquid Chromatography (HPLC) pump, an autosampler or a valving system with sample loops, and a fraction collector. All systems, whether automated or manual, must meet the calibration requirements of Section 10.3.3.

NOTE: GPC cleanup is required for all soil/sediment extracts, and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target analytes.

6.2.6.2 Chromatographic Column - 700 mm x 25 mm ID glass column. Flow is upward. To simplify switching from the ultraviolet (UV) detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

6.2.6.3 Guard Column (optional) - 5 cm, with appropriate fittings to connect the inlet side of the analytical column.

6.2.6.4 Bio Beads (SX-3) - 200-400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, or equivalent). An additional 5 g of Bio Beads are required if the optional guard column is employed. The quality of Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they can also pass through the column screens and damage the valve.

6.2.6.5 Ultraviolet Detector - Fixed wavelength (254 nm) with a semi-prep flow-through cell.

6.2.6.6 Strip Chart Recorder - Recording integrator or laboratory data system.

6.2.6.7 Syringe Filter Assembly, disposable - 5 micron filter discs.

NOTE: Consult the instrument operation manual to determine the proper filter disc to use in the system. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

6.2.6.8 Viscometer

6.2.7 Kuderna-Danish (K-D) Apparatus

6.2.7.1 Concentrator Tubes - 15 mL and 10 mL, graduated. Ground-glass stoppers are used to prevent evaporation of extracts.

6.2.7.2 Evaporative Flasks - 500 mL.

6.2.7.3 Silicon Carbide Boiling Chips - Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride. PTFE boiling chips solvent rinsed prior to use are acceptable.

6.2.7.4 Snyder Column - Three-ball macro.

6.2.7.5 Snyder Column - Two-ball micro.

6.2.8 Nitrogen Evaporation Device - Equipped with a water bath that can be maintained at 35-40°C. To prevent the release of solvent fumes into the laboratory, the nitrogen evaporator device must be used in a hood.

6.2.9 Pressurized Fluid Extraction Device

6.2.9.1 Dionex Accelerated Solvent Extractor (ASE-300) or equivalent with appropriately sized extraction cells. Currently, 100 mL cells are available that will accommodate greater than 30 g samples. Cells should be made of stainless steel or other material capable of withstanding the pressure environments [2000+ pounds per square inch (psi)] necessary for this procedure.

6.2.9.2 Other system designs may be employed, provided that adequate performance can be demonstrated for the analytes and matrices of interest.

6.2.10 Sonabox Acoustic Enclosure (or equivalent) - For use with disrupter to decrease noise level.

6.2.11 Ultrasonic Cell Disruptors - QSonica LLC, (53 Church Hill Road, Newtown, CT 06470) model S-4000 or equivalent ultrasonic liquid disruptor – equipped with a 3/4-inch horn and a 1/2-inch tapered horn, and a 1/8-inch standard tapered microtip probe with a minimum output capacity of 300 watts.

NOTE 1: To ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. A rough tip surface is an indication of erosion.

NOTE 2: Follow manufacturer’s instructions for set-up.

6.2.12 Vacuum Filtration Apparatus

6.2.12.1 Buchner Funnel.

6.2.12.2 Filter Paper - Whatman No. 42 or equivalent.

6.2.13 Water Bath - Heated, with concentric ring cover, capable of temperature control. The bath should be used in a hood.

6.3 Analytical Instrumentation

6.3.1 Gas Chromatograph

The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants, or flow controllers with rubber components, are not to be used.

6.3.2 Gas Chromatography Columns

Recommended Columns: Minimum length 30 meter (m) x 0.25 mm ID (or 0.32 mm) bonded-phase fused silica capillary column DB-5 (J&W Scientific); Rtx®-5, Rtx®-5 Sil Ms (Restek); Zebron ZB-5 (Phenomenex); SPB-5 (Supelco); AT-5 (Alltech); HP-5 (Agilent); CP-Sil 8CB (Chrompack); 007-2 (Quadrex); BP-5 (SGE); or equivalent. A description of the GC column used for analysis shall be provided in the SDG Narrative. Packed columns cannot be used.

6.3.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants that interfere with the identification and quantitation of the analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated using the column meet the initial calibration, initial calibration verification (ICV), and continuing calibration verification (CCV) technical acceptance criteria (Sections 9.3.5, 9.4.5, and 9.5.5) and the CRQLs listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits;
- The column must be capable of accepting up to 160 ng of each analyte listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits, without becoming overloaded; and
- The column provides equal or better resolution of the analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits, than the columns listed in Section 6.3.2.

6.3.2.1.1 As applicable, follow the manufacturer's instructions for use of its product.

6.3.2.1.2 The Contractor must maintain documentation that the column meets the criteria in Section 6.3.2.1. The minimum documentation is as follows:

6.3.2.1.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.3.2.1.2.2 RICs and data system reports generated on the GC/MS used for EPA Contract Laboratory Program (CLP) analyses:

- From method blanks that demonstrate that there are no contaminants that interfere with the semivolatile analysis when using the alternate column; and
- From initial calibration, ICV, and CCV standards analyzed using the alternate column.

6.3.2.1.3 Based on the Contractor-generated data described above, the Contractor must complete a written review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Sections 9.3.5, 9.4.5, and 9.5.5;
- The low-point initial calibration standard analysis has adequate sensitivity to meet the semivolatile CRQLs;
- The high-point initial calibration standard analysis was not overloaded; and

- The column does not introduce contaminants that interfere with the identification and/or quantitation of analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits.

6.3.2.1.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request by the EPA Regional CLP Contracting Officer's Representative (COR).

6.3.3 Mass Spectrometer

The MS must be capable of scanning from 35-500 atomic mass units (u) every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the decafluorotriphenylphosphine (DFTPP) GC/MS performance check technical acceptance criteria in Table 2 – Decafluorotriphenylphosphine Key Ions and Ion Abundance Criteria, when 50 ng of DFTPP is injected through the GC inlet. The system must be capable of SIM. The Contractor is to use professional judgment and the instrument manufacturer's instructions and guidelines in choosing an appropriate single ion scan or dwell time (usually 50-500 msec per ion).

The instrument must be vented to the outside of the facility or to a trapping system that prevents the release of contaminants into the instrument room.

6.3.4 Gas Chromatograph/Mass Spectrometer Interface

Any GC/MS interface may be used that gives acceptable sensitivity at the CRQLs. However, direct insertion of the GC column into the MS ion source is the recommended interface. GC/MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

6.4 Data Systems/Data Storage

A computer system must be interfaced to the MS that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching of any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an EICP. Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows comparing sample spectra against reference library spectra. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

7.0 REAGENTS AND STANDARDS

The Contractor must provide all standards to be used with the contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Organic Analytical Methods, Section 11. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1 Reagents

7.1.1 Reagent Water - Reagent water is defined as water in which an interferent is not observed at or above the CRQL for each analyte of interest.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g [1 pound (lb)] of activated carbon.

7.1.1.2 Reagent water may also be generated using a water purification system.

7.1.2 Acetone/methylene chloride (1:1 v/v).

7.1.3 Hydromatrix™ - Diatomaceous earth-based material rinsed with methylene chloride and dried at 400°C for 4 hours in a shallow tray, cooled in a desiccator, and stored in a glass bottle.

7.1.4 Sodium sulfate - Granular anhydrous reagent grade, heated at 400°C for 4 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by a GC/Electron Capture Detector (ECD) to demonstrate that it is free of interference before use or must be purchased with certification that it is free of interference.

CAUTION: AN OPEN CONTAINER OF SODIUM SULFATE MAY BECOME CONTAMINATED DURING STORAGE IN THE LABORATORY.

7.1.5 Solvents: Acetone, methanol, methylene chloride, iso-octane, 2-propanol, and toluene - pesticide residue analysis grade or equivalent.

7.1.6 Sulfuric acid, concentrated, 95-98% (sp. gr. 1.84).

7.1.7 Glycerol.

7.2 Standards

7.2.1 Stock Standard Solutions

7.2.1.1 Stock standard solutions are defined as standards that are to be used to produce working standards. They may be in the form of single compounds or mixtures. They may be prepared in methylene chloride from pure standard materials, or purchased as pre-made solutions. Prepare fresh stock standards every 6 months, or sooner if the standard has degraded or evaporated.

7.2.2 Working Standards

7.2.2.1 Initial and Continuing Calibration Solutions

7.2.2.1.1 Prepare the calibration standards at a minimum of five concentrations in methylene chloride that are applicable to the sensitivity of the instrument. For most operations, the calibration standards are to be prepared at 5.0, 10, 20, 40, and 80 ng/µL for each target analyte and associated DMCs (see Table 3 – Semivolatile Deuterated Monitoring Compounds and the Associated Target Analytes and Table 4 – Semivolatile Deuterated Monitoring Compounds and the Associated Target Analytes for Optional Analysis by Selected Ion Monitoring), except 1,4-Dioxane and target analytes in Section 7.2.2.1.2. For 1,4-Dioxane and 1,4-Dioxane-d8, the calibration standard concentrations shall be at 2.0, 4.0, 8.0, 16, and 32 ng/uL. These levels are based upon 1.0 mL final volume extracts for samples not undergoing GPC cleanup, and 0.5 mL final volume extracts for those samples undergoing GPC cleanup. Other concentrations may be used for more sensitive instrumentation and final extract volumes. For example, a laboratory may use a final extract volume of 1.0 mL for samples undergoing GPC cleanup, and a low calibration standard of 2.5 ng/µL. The alternate calibration standards and final volumes may be used as long as the following requirements are met:

7.2.2.1.1.1 The laboratory can demonstrate that the CRQL for each analyte listed in Exhibit C –Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits can be reached using the calibration and final volume scheme. This demonstration is made when there is formal documentation of laboratory Method Detection Limit (MDL) studies indicating that the calculated MDL for each target analyte is below the required CRQL for that analyte when using the laboratory's specific final volume and calibration level scheme.

7.2.2.1.1.2 All five calibration levels are in the same ratio as that shown above (e.g., if a laboratory were using a 1.0 ng/µL low standard, then the other calibration levels must be 2.0, 4.0, 8.0, and 16 ng/µL).

7.2.2.1.2 Each calibration standard should contain each target analyte. Each DMC may be added to the other calibration standards, or may be contained in a separate mixture and combined with the calibration standard in the autosampler vials just prior to analysis. Twenty-one target analytes and six DMCs [Benzaldehyde, Phenol, Bis(2-chloroethyl) ether, 2-Methylphenol, 2,2'-Oxybis(1—chloropropane), Acetophenone, 4-Chloroaniline, Caprolactam, Hexachlorocyclopentadiene, Atrazine, Carbazole, Fluoranthene, 3,3'-Dichlorobenzidine, Di-n-octylphthalate, 2,4-Dinitrophenol, PCP, 4-Methylphenol, 4,6-Dinitro-2-methylphenol, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, Phenol-d_5, Bis(2-chloroethyl) ether-d_8, 4-Methylphenol-d_8, 4-Chloroaniline-d_4, 4-Nitrophenol-d_4, and 4,6-Dinitro-2-methylphenol-d_2] have shown to be less sensitive. These analytes will require a five-point initial calibration at 10, 20, 40, 80, and 160 ng/µL.

NOTE: 1.0 or 2.0 µL injections of all calibration standards may be used. All samples analyzed must have been injected at the same volume (1.0 or 2.0 µL) as the calibration standard.

7.2.2.1.2.1 For PAHs and PCP only full scan analyses, the initial calibration containing all target analytes and DMCs can be used to substitute the five-point initial calibration containing only these target analytes and associated DMCs at the concentrations in Sections 7.2.2.1 and 7.2.2.5.

7.2.2.1.2.2 If the optional analysis of PAHs and PCP using the SIM technique is to be performed, prepare calibration standards at a minimum of five concentration levels that are applicable to the sensitivity of the instrument. For most operations, the calibration standards are to be prepared at 0.10, 0.20, 0.40, 0.80, and 1.6 ng/µL for each target analyte of interest and the associated DMCs (see Table 10 – Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation of Polynuclear Aromatic Hydrocarbon and Pentachlorophenol). PCP will require a five-point initial calibration at 0.20, 0.40, 0.80, 1.6, and 3.2 ng/µL.

NOTE: 1.0 or 2.0 µL injections of all calibration standards may be used. All samples analyzed must have been injected at the same volume (1.0 or 2.0 µL) as the calibration standard.

7.2.2.1.3 The CCV standard should be at or near the mid-point concentration level of the calibration standards. If the optional analysis of PAHs and PCP by SIM is to be performed, the CCV standard should be at or near the mid-point calibration level, normally 0.40 ng/µL (0.80 ng/µL for PCP).

7.2.2.1.4 To facilitate the confirmation of single component pesticides from the semivolatile library search data (see Exhibit D – Pesticides Analysis, Section 11.1.2), the laboratory may include the single component pesticide target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits in the semivolatile CCV standard. The laboratory may add any or all of these analytes to the semivolatile CCV standard, but at a concentration of 10 ng/µL or less. Do not include the Aroclor or Toxaphene mixtures in the semivolatile initial and CCV standards. If added to this standard, these additional analytes must be included in the quantitation report for the CCV standard. As only a single point calibration would be performed, no Percent Relative Standard Deviation (%RSD) or Percent Difference (%D) criteria would apply to these additional analytes.

7.2.2.2 Initial Calibration Verification Solution

Prepare the working initial calibration standard solution containing all of the target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte and Contract Required Quantitation Limits) from an alternate source or a different lot than that used for the initial calibration (ICAL) standard analyses in methylene chloride. Prepare a fresh calibration standard

solution every month, or sooner if the solution has degraded or evaporated.

7.2.2.2.1 The ICV standard shall be at a concentration equivalent to the mid-level calibration standards. If the optional analysis of PAHs and PCP by SIM is to be performed, the ICV standard should be at or near the mid-point calibration level, normally 0.40 ng/µL (0.80 ng/µL for PCP).

7.2.2.2.2 The ICV standard shall be prepared by the same procedures as the CCVs.

7.2.2.3 Instrument Performance Check Solution

Prepare a solution containing DFTPP in methylene chloride. The solution may be incorporated into the calibration standard used as the mid-level initial calibration standard and the CCV standard, or may be prepared as a single compound solution. If DFTPP is incorporated into the calibration standard, then an aliquot of the DFTPP solution is to be added to the autosampler vial containing either the initial calibration mid-level standard or the CCV standard before calibration analysis. The DFTPP must be analyzed using the same GC and MS analytical conditions as is used for the calibration analysis. The DFTPP solutions are to be prepared such that 50 ng of DFTPP is injected onto the column.

7.2.2.4 Gel Permeation Chromatography Calibration Solution

Prepare a GPC calibration solution in methylene chloride containing the following analytes at the minimum concentration listed (in elution order). The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

Compound	Concentration (mg/mL)
Corn oil (CAS# 8001-30-7)	25.0
Bis(2-ethylhexyl)phthalate (CAS# 117-81-7)	0.5
Methoxychlor (CAS# 72-43-5)	0.1
Perylene (CAS# 198-55-0)	0.02
Sulfur (CAS# 7704-34-9)	0.08

NOTE: Sulfur is not very soluble in methylene chloride, but it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds.

7.2.2.5 Deuterated Monitoring Compound Spiking Solution

7.2.2.5.1 Prepare a DMC spiking solution containing the following DMC analytes in methanol at the concentration given:

DMC	Concentration µg/mL
1,4-Dioxane-d_8	16
Phenol-d_5	80
Bis(2-chloroethyl)ether-d_8	80
2-Chlorophenol-d_4	80
4-Methylphenol-d_8	80
4-Chloroaniline-d_4	80
Nitrobenzene-d_5	80

DMC	Concentration µg/mL
2-Nitrophenol-d_4	80
2,4-Dichlorophenol-d_3	80
Dimethylphthalate-d_6	80
Acenaphthylene-d_8	80
4-Nitrophenol-d_4	80
Fluorene-d_{10}	80
4,6-Dinitro-methylphenol-d_2	80
Anthracene-d_{10}	80
Pyrene-d_{10}	80
Benzo(a)pyrene-d_{12}	80
Fluoranthene-d_{10} (SIM analysis)	0.8
2-Methylnaphthalene-d_{10} (SIM analysis)	0.8

7.2.2.5.2 DMC spiking solution is added (500 µL) prior to sample processing to all samples, blanks, requested Matrix Spike and Matrix Spike Duplicates (MS/MSDs), and calibration solutions.

7.2.2.5.3 The SIM DMC compounds (Table 4 – Semivolatile Deuterated Monitoring Compounds and the Associated Target Analytes for Optional Analysis by Selected Ion Monitoring) can be added as part of the DMC spiking solution or added separately to all standards, samples, and blanks that require SIM analysis.

7.2.2.5.4 The DMC spiking solution must be prepared every 12 months, or sooner if the solution has degraded or concentrated.

7.2.2.6 Matrix Spiking Solution

7.2.2.6.1 If MS/MSD analysis is requested at the time of scheduling, prepare a spiking solution in methanol that contains the following analytes and concentrations:

Bases/Neutrals	µg/mL	Acids	µg/mL
Acenaphthene	80	Pentachlorophenol	80
2,4-Dinitrotoluene	80	Phenol	80
Pyrene	80	2-Chlorophenol	80
N-Nitroso-di-n-propylamine	80	4-Chloro-3-methylphenol	80
		4-Nitrophenol	80

7.2.2.6.2 For the analysis of PAH and PCP-only, the laboratory has the option of using the matrix spiking solution in Section 7.2.2.6.1 or preparing a matrix spiking solution containing only acenaphthene, pyrene, and PCP at the concentration of 80 µg/mL in methanol for full scan or 0.80 µg/mL for SIM analysis.

7.2.2.7 Internal Standard Spiking Solution

7.2.2.7.1 Prepare an internal standard spiking solution containing each of the following compounds in methylene chloride: 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. It may be necessary to use 5-10% toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. Just prior to full scan analysis by GC/MS, add sufficient amount of the internal standard spiking solution to an aliquot of the water, low-level, or medium-level soil

sample extracts for the initial analysis, dilution, and reanalysis, or to the re-extracts if applicable, to result in a 20 ng/µL concentration of each internal standard. Prepare a fresh internal standard spiking solution monthly, or sooner if the solution has degraded or concentrated.

7.2.2.7.2 If the optional analysis of PAHs and PCP using the SIM analysis is to be performed, the Contractor shall add sufficient amount of the internal standard spiking solution to an aliquot of the water or low-level sample extracts for the initial analysis, dilution, and reanalysis, or to the re-extracts if applicable, just prior to SIM analysis to result in a 0.40 ng/µL concentration of each internal standard. 1,4-dichlorobenzene-d_4 is not required to be evaluated as internal standard when performing SIM analysis.

7.2.3 Storage of Standard Solutions

7.2.3.1 Store the working standards at ≤6°C, but not frozen, in PTFE-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after 6 months, or sooner if comparison with quality control (QC) check samples indicates a problem.

7.2.3.2 Store the stock standard solutions at ≤6°C, but not frozen, in PTFE-lined screw-cap amber bottles.

7.2.3.3 Standard solutions purchased from a chemical supply company as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer's expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor that are immediately ampulated in glass vials may be retained for 2 years from the preparation date. The expiration date of the ampulated standards, upon the breaking of the glass seal, is 6 months (or sooner if the standard has degraded or evaporated).

7.2.3.4 Refrigeration of the GPC calibration solution may cause the corn oil to precipitate. Before use, allow the solution to stand at room temperature until the corn oil dissolves. Replace this calibration solution every 6 months, or more frequently if necessary.

7.2.3.5 Protect all standards from light.

7.2.3.6 Samples, sample extracts, and standards must be stored separately.

7.2.3.7 The Contractor is responsible for maintaining and verifying the integrity of standard solutions prior to use. This means, at a minimum, the standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution.

7.2.4 Temperature Records for Storage of Standards

7.2.4.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators/freezers.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Water Samples

Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with PTFE-lined screw-caps. If amber containers are not available, the samples should be protected from light.

8.1.2 Soil/Sediment Samples

Soil/sediment samples may be collected in glass containers.

8.2 Procedure for Sample and Sample Extract Storage

8.2.1 Sample Storage

The samples must be protected from light and refrigerated at ≤6°C, but not frozen, from the time of receipt until 60 days after delivery of a complete, reconciled data package to the EPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 Sample Extract Storage

Sample extracts must be protected from light and stored at ≤6°C, but not frozen, until 365 days after delivery of a complete, reconciled data package to the EPA.

8.3 Contract Required Holding Times

8.3.1 Extraction of water samples shall be started within 5 days of Validated Time of Sample Receipt (VTSR). Extraction of TCLP or SPLP leachates shall begin within 7 days from the completion of the leaching procedure. Extraction of soil/sediment samples shall be completed within 10 days of VTSR.

8.3.2 Analysis of sample extracts must be analyzed within 40 days following the start of extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Initial Instrument Set-Up

9.1.1 Gas Chromatograph

9.1.1.1 The recommended GC analytical conditions are provided in Table 6 – Gas Chromatograph Analytical Conditions. Other conditions may be used, provided that all technical acceptance criteria in Sections 9.3.5, 9.4.5, 9.5.5, and 11.3 are met. For example, newer columns that are stable at temperatures of up to 370°C may be used. The use of these columns would decrease analysis time while still providing adequate resolution.

9.1.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSDs.

9.1.1.3 The same injection volume, 1.0 or 2.0 µL, must be used for all standards, samples (including MS/MSDs and required method blanks).

9.1.2 Mass Spectrometer

The recommended MS analytical conditions are provided in Table 7 – Mass Spectrometer Analytical Conditions.

9.2 Instrument Performance Check

9.2.1 Summary of GC/MS Instrument Performance Check

9.2.1.1 The GC/MS system must be tuned to meet the manufacturer's specifications using a suitable calibrant such as perfluoro-tri-n-butylamine (FC-43) or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Section 7.2.2.3).

9.2.1.2 Prior to the analysis of any samples, including MS/MSDs, blanks, or calibration standards, the Contractor must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing DFTPP.

9.2.1.3 The requirement to analyze the instrument performance check solution is optional when SIM analysis of PAHs and PCP is to be performed.

9.2.2 Frequency of GC/MS Instrument Performance Check

The instrument performance check solution must be injected once at the beginning of each 12-hour period during which samples, blanks, or standards are analyzed. The 12-hour period for the GC/MS instrument performance check, calibration standards (initial calibration, ICV, or CCV), blank, and sample analysis begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant instrument performance check. However, in cases where a closing CCV can be used as an opening CCV for the next 12-hour period, then an additional DFTPP tune is not required, and the 12-hour period begins with the injection of the CCV. The period ends after 12 hours have elapsed according to the system clock.

NOTE: For the optional analysis of PAHs and PCP by the SIM technique, the 12-hour period begins at the moment of injection of the first initial calibration standard or at the moment of injection of the CCV standard, if initial calibration is not to be performed. The time period ends after 12 hours have elapsed according to the system clock.

9.2.3 Procedure for GC/MS Instrument Performance Check

The analysis of the instrument performance check solution shall be performed as follows:

- As an injection of 50 ng of DFTPP into the GC/MS.
- By adding a sufficient amount of DFTPP to the mid-level calibration standard to result in an on-column amount of 50 ng of DFTPP (Section 7.2.2.3).

9.2.4 Technical Acceptance Criteria for GC/MS Instrument Performance Check

9.2.4.1 The GC/MS system must be tuned at the frequency described in Section 9.2.2.

9.2.4.2 The abundance criteria listed in Table 2 – Decafluorotriphenylphosphine Key Ions and Ion Abundance Criteria, must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP must be acquired in the following manner:

9.2.4.2.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

9.2.4.2.2 Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. Do not background subtract part of the DFTPP peak.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must be analyzed under identical GC/MS instrument analytical conditions.

9.2.4.3 The chromatographic resolution of the GC system must be capable of resolving the structural isomers Benzo[b] and Benzo[k]fluoranthene. The chromatographic resolution of the GC system must show a minimum 50% valley between Benzo[b] and Benzo[k]fluoranthene (i.e., the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights).

9.2.5 Corrective Action for GC/MS Instrument Performance Check

9.2.5.1 If the DFTPP technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source or take other corrective actions to achieve the technical acceptance criteria.

9.2.5.2 Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to the analysis of samples (including MS/MSDs) and required blanks, and after the instrument performance check technical acceptance criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations (Section 7.2.2.1.1) to determine instrument sensitivity and the linearity of GC/MS

response for the semivolatile target analytes and DMCs. For PAHs and PCP only full scan analysis, initial calibration shall be performed for these target analytes and their associated DMCs at the required concentration levels (see Section 7.2.2.1.2.2).

NOTE: For optional analysis of PAHs and PCP only using the SIM technique, the GC/MS system must be calibrated at a minimum of five concentrations (Section 7.2.2.1.2.2), prior to the analysis of samples and required blanks, to determine instrument sensitivity and linearity.

9.3.2 Frequency of Initial Calibration

9.3.2.1 Each GC/MS system must be calibrated prior to analyzing samples, whenever the Contractor takes corrective action that may change or affect the initial calibration criteria (e.g., ion source cleaning or repairs, column replacement, etc.), or if the CCV technical acceptance criteria have not been met.

9.3.2.2 If time remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration, samples and blanks may be analyzed (Section 9.3.5). It is not necessary to analyze another CCV standard. A method blank is required.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set up the GC/MS system as described in Section 9.1.

9.3.3.2 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.3.3.3 Add internal standard spiking solution (Section 7.2.2.7) to aliquots of each of the five calibration standards to result in a 20 ng/µL concentration of each internal standard. The internal standards specified in Section 7.2.2.7 should permit most of the semivolatile target analytes to have Relative Retention Times (RRTs) of 0.80 to 1.20, using the assignments of internal standards to target analytes given in Table 9 – Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation.

9.3.3.4 Analyze each calibration standard by injecting 1.0 or 2.0 µL of standard. The initial calibration sequence is listed below.

INITIAL CALIBRATION SEQUENCE

1. GC/MS Instrument Performance Check
2. CS1 Initial Calibration Standard
3. CS2 Initial Calibration Standard
4. CS3 Initial Calibration Standard
5. CS4 Initial Calibration Standard
6. CS5 Initial Calibration Standard

9.3.4 Calculations for Initial Calibration

9.3.4.1 Calculate the RRF for each semivolatile target analyte and DMC using Equation 1. The primary characteristic ions used for quantitation are listed in Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards. Assign the target analytes and DMCs to an internal standard according to Table 9 - Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation and Table 10 – Internal Standards with Associated Target and Deuterated Monitoring

Compounds Assigned for Quantitation of Polynuclear Aromatic Hydrocarbon and Pentachlorophenol. For internal standards, use the primary ion listed in Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards unless interferences are present. If interferences prevent the use of the primary ion for a given internal standard, use the secondary ion(s) listed in Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards.

NOTE: Unless otherwise stated, the area response of the primary characteristic ion is the quantitation ion.

EQ. 1 Relative Response Factor

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

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards)

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards). The target analytes are listed with their associated internal standards in Table 9 - Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation.

C_{is} = Amount of the internal standard injected (ng)

C_x = Amount of the target analyte or DMC injected (ng)

9.3.4.2 The Mean RRF ($\overline{RRF}$) must be calculated for all compounds according to Equation 2.

9.3.4.3 Calculate the %RSD of the RRF values for each target analyte and DMC over the initial calibration using Equation 3 in conjunction with Equations 2 and 4.

9.3.4.3.1 Equation 2 is the general formula for the mean of a set of values.

EQ. 2 Mean Value

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

WHERE,

X_i = Value

$\overline{X}$ = Mean value

n = Number of values

9.3.4.3.2 Equation 3 is the general formula for the relative standard deviation.

EQ. 3 Percent Relative Standard Deviation

$$\%RSD = \frac{SD_{RRF}}{\overline{X}} \times 100$$

WHERE,

SD_{RRF} = Standard deviation of initial calibration RRFs (per compound) from EQ. 4

$\bar{x}$ = Mean value of the initial calibration RRFs (per compound)

9.3.4.3.3 Equation 4 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 4 Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{(n-1)}}$$

WHERE,

X_i = Each individual value used to calculate the mean

$\bar{x}$ = The mean of n values

n = Total number of values

9.3.5 Technical Acceptance Criteria for Initial Calibration

9.3.5.1 All initial calibration standards must be analyzed at the concentrations described in Section 7.2.2.1.1 and at the frequency described in Section 9.3.2 on a GC/MS system meeting the DFTPP technical acceptance criteria (Section 9.2.4).

9.3.5.2 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

NOTE: The EPA Regional customer may specify, at the time of scheduling, that certain analytes of interest (i.e., PCP) may not fail the performance criteria.

9.3.5.3 The RRF at each calibration concentration for each semivolatile target analyte and DMC must be greater than or equal to the compound's minimum RRF listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.3.5.4 The %RSD for each target analyte and DMC listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds must be less than or equal to the value listed.

9.3.5.5 Up to four target analytes and DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to four target analytes and DMCs (excluding those with maximum %RSD requirements of 40.0%) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds, but these compounds must still meet the maximum %RSD requirements of 40.0%.

9.3.5.6 For the optional analysis of PAHs and PCP either by full scan or using the SIM technique, two target analytes and DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to two target analytes and DMCs (excluding those with maximum %RSD requirements of 40.0%) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds, but these compounds must still meet the maximum %RSD requirements of 40.0%.

9.3.5.7 The chromatographic resolution should be verified with the mid-point concentration of the initial calibration as well as the CCV standards if closely eluting isomers are to be reported. Sufficient chromatographic resolution is achieved when the height of the valley between the two isomer peaks is less than 50% of the average of the two peak heights.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the acceptance criteria.

9.3.6.2 Any samples or required blanks analyzed when initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.4 Initial Calibration Verification

9.4.1 Summary of Initial Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check and initial calibration technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an ICV (containing all the target analytes from an alternate source or a different lot than the ICAL standards, and the DMCs and internal standards from the same source or lot as in the ICAL standards) to ensure that the instrument is calibrated accurately.

9.4.2 Frequency of Initial Calibration Verification

The calibration for each GC/MS system used for analysis must be verified with an ICV at the frequency of one per ICAL analytical sequence. The ICV shall be analyzed following that last ICAL standard analysis and prior to any method blank, sample, or applicable CCV analysis.

Injection #	**Material Injected**
1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	BFB then CS1–CS5 First 6 steps of the initial calibration
7th - ICV	ICV
8th - blanks, samples, MS/MSDs	Blanks, samples, and MS/MSDs
9th - Subsequent Samples	

9.4.3 Procedure for Initial Calibration Verification

9.4.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.4.3.2 Add sufficient amount of internal standard solution (Section 7.2.2.7) to the ICV (Section 7.2.2.2) and the DMC solution (Section 7.2.2.5).

9.4.3.3 Analyze the ICV standard according to Section 10.4 using the same injection volume as in the initial calibration.

9.4.4 Calculations for Initial Calibration Verification

9.4.4.1 Calculate an RRF for each target analyte and DMC according to Section 9.3.4.1.

9.4.4.2 Calculate the %D between the ICV RRF_c and the preceding initial calibration $\overline{RRF_i}$ for each target analyte and DMC using the following equation:

EQ. 5 Initial Calibration Verification Percent Difference

$$\%D = \frac{RRF_C - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

WHERE,

RRF_c = Relative Response Factor from current ICV standard

$\overline{RRF_i}$ = Mean Relative Response Factor from the preceding initial calibration

9.4.5 Technical Acceptance Criteria for Initial Calibration Verification

9.4.5.1 The concentration of the semivolatile target analytes and DMCs in the ICV must be at or near the mid-point concentration of the calibration standards. The ICV must be analyzed at the frequency described in Section 9.4.2, on a GC/MS system meeting the DFTPP (Section 9.2.4) and the initial calibration (Section 9.3.5) technical acceptance criteria. For the optional analysis of PAHs and PCP using only SIM, the ICV must be analyzed at or near the mid-point concentration level of the calibration range, 0.40 ng/µL (0.80 ng/µL for PCP), at the frequency described in Section 9.4.2, and on a GC/MS system meeting the initial calibration technical acceptance criteria.

9.4.5.2 For an ICV, the RRF for each target analyte and DMC must be greater than, or equal to, the compound's minimum RRF listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to four target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.4.5.3 For the ICV of the optional analysis of PAHs and PCP using the full scan method or the SIM technique, up to two target analytes and DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.4.5.4 For an ICV, the %D for each target analyte and DMC must be less than, or equal to, the compound's maximum %D listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to four target analytes and DMCs (excluding those with maximum %D requirements of ±40%) may fail to meet the %D criteria listed in Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.4.5.5 For the ICV of the optional analysis of PAHs and PCP using the full scan method or the SIM technique, up to two target analytes and DMCs (excluding those with maximum %D requirements of ±40%) may fail to meet the %D criteria listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.4.5.6 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.4.6 Corrective Action for Initial Calibration Verification

9.4.6.1 If the ICV technical acceptance criteria are not met, recalibrate the GC/MS instrument according to Section 9.3.

9.4.6.2 If the ICV fails to meet the technical acceptance criteria and a subsequent reanalysis of the ICV meets the technical acceptance criteria, proceed to the blank and sample analyses. All sample and required blank analyses must be associated to a compliant ICV analysis following the associated ICAL.

9.5 Continuing Calibration Verification

9.5.1 Summary of Continuing Calibration Verification

Prior to the analysis of samples and required blanks, and after instrument performance check, initial calibration, and ICV technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing an opening CCV (containing all the semivolatile target analytes, DMCs, and internal standards) to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the analytical method. A closing CCV using the same standard conditions as for the opening CCV is required after all samples and blanks have been analyzed, and before the end of the 12-hour period (refer to the analytical sequence in Section 9.5.2.3). If the closing CCV meets opening CCV criteria, an additional DFTPP time is not required and the next 12-hour period begins with this CCV.

9.5.2 Frequency of Continuing Calibration Verification

9.5.2.1 The calibration for each GC/MS system used for analysis must be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the injection of DFTPP for full scan analysis, followed by the injection of the opening CCV. If a closing CCV meets the technical acceptance criteria for an opening CCV (Section 9.5.5) and samples are analyzed within the next 12-hour period, then an additional DFTPP tune is not required and the 12-hour period begins with that calibration verification. If the closing CCV does not meet the technical

acceptance criteria for an opening CCV, then a DFTPP tune, followed by an opening CCV, is required and the next 12-hour period begins with the DFTPP tune (Section 9.2.2).

9.5.2.2 If time still remains in the 12-hour period after meeting the technical acceptance criteria for the initial calibration and ICV, samples may be analyzed.

9.5.2.3 After the injection of all samples and required blanks, and before the end of the 12-hour period, another injection of the CCV solution is required (closing CCV). The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence, provided that all technical acceptance criteria are met for an opening CCV in Section 9.5.5.

Time	**Injection #**	**Material Injected**
0 hr	1st - 6th - GC/MS Instrument Performance Check followed by CS1 – CS5 calibration standards	DFTPP then CS1–CS5 First 6 steps of the initial calibration
	7th - ICV	ICV
	8th - blanks, samples, MS/MSDs	Blanks, samples, MS/MSDs
	9th - Subsequent Samples	
End 12 hr	Closing CCV (meeting Closing CCV criteria, but not opening CCV)	CS3 – Closing CCV
New 12 hr	1st GC/MS Instrument Performance Check	DFTPP Instrument Performance Check
	2nd - injection past 12 hours Opening CCV	CS3 - Opening CCV
		Blank, MS/MSD, subsequent samples Subsequent Samples Last Sample
End 12 hr	Closing CCV (meeting Closing CCV criteria but not Opening CCV)	CS3 – Closing CCV
New 12 hr	1st injection Instrument Performance Check	DFTPP Instrument Performance Check
	2nd Injection Opening CCV	CS3 – Opening CCV
		Blank, MS/MSD, subsequent samples Subsequent Samples Last Sample
End of 12 hr beginning of next 12 hr	Closing CCV (meeting Opening CCV criteria) Instrument Performance Check not required	CS3 – Closing CCV meeting Opening CCV
		Blank, MS/MSD, subsequent samples Subsequent Samples Last Sample
End of 12 hr	Closing CCV meeting criteria	CS3 – Closing CCV meeting Opening CCV

NOTE: For analysis using the SIM technique, prior to the analysis of samples and required blanks, and after initial calibration technical acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a CCV standard.

9.5.3 Procedure for Continuing Calibration Verification

9.5.3.1 All standard/spiking solutions must be allowed to warm to ambient temperature before analysis.

9.5.3.2 Add internal standard spiking solution (Section 7.2.2.7) to an aliquot of CCV standard to result in 20 ng/µL concentration of each internal standard. The internal standards specified in Section 7.2.2.7 should permit most of the semivolatile target analytes to have RRTs of 0.80-1.20, using the assignments of internal standards to target analytes given in Table 9 – Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation.

9.5.3.3 Analyze the CCV standard according to Section 10.4 using the same injection volume as in the initial calibration.

9.5.4 Calculations for Continuing Calibration Verification

9.5.4.1 Calculate an RRF for each semivolatile target analyte and DMC according to Section 9.3.4.1.

9.5.4.2 Calculate the %D between the CCV RRF_c and the most recent initial calibration $\overline{RRF}_i$ for each semivolatile target analyte and DMC using the following equation:

EQ. 6 Internal Standard Calibration Percent Difference

$$\%D = \frac{RRF_c - \overline{RRF}_i}{\overline{RRF}_i} \times 100$$

WHERE,

RRF_c = Relative Response Factor from current CCV standard

$\overline{RRF}_i$ = Mean Relative Response Factor from the most recent initial calibration

9.5.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.5.5.1 The concentration of the semivolatile target analytes and DMCs in the opening and closing CCV must be at or near the mid-point concentration of the calibration standards. The opening and closing CCV standard must be analyzed at the frequency described in Section 9.5.2, on a GC/MS system meeting the DFTPP (Section 9.2.4), initial calibration (Section 9.3.5), and ICV (Section 9.4.5) technical acceptance criteria.

NOTE: For the optional analysis of PAHs and PCP using only SIM, the opening and closing CCV must be analyzed at or near the mid-point concentration level of the calibration range, 0.40 ng/µL (0.80 ng/µL for PCP), at the frequency described in Section 9.5.2, and on a GC/MS system meeting the initial calibration technical acceptance criteria.

9.5.5.2 For an opening or closing CCV, the RRF for each semivolatile target analyte and DMC must be greater than, or equal to, the compound's opening or closing minimum RRF listed in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

9.5.5.3 For an closing CCV, the %D for each semivolatile target analyte and DMC must be in the inclusive range of the compound's %D value in Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds, with the exception of the following eleven target analytes: 4-Chloroaniline, Pentachlorophenol, 2,4-Dinitrophenol, Benzo(a)pyrene, Benzo[b]fluoranthene, Benzo[k]fluoranthene, Benzo[g,h,i]perylene, Di-n-octylphthalate, Dibenzo[a,h]anthracene, Hexachlorocyclopentadiene, and Indeno(1,2,3-cd)pyrene. The closing CCV RRF %D requirement is advisory for these eleven target analytes.

9.5.5.4 For the opening CCV of the optional analysis of PAHs, PCP, and DMC using SIM, the %D must be within the inclusive range of the compound's %D value listed in Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. For a closing CCV, the %D must be within the inclusive range of the compound's %D value listed, with the exception of the following seven target analytes: Benzo(a)pyrene, Benzo[b]fluoranthene, Benzo[k]fluoranthene, Benzo[g,h,i]perylene, Dibenzo[a,h]anthracene, Indeno(1,2,3-cd)pyrene, and PCP. The closing CCV RRF %D requirement for analysis by SIM is advisory for these seven target analytes.

9.5.5.5 For an opening or closing CCV, up to four target analytes and/or DMCs (excluding those compounds with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to four target analytes and/or DMCs (excluding those with maximum %D requirements of ±40.0%) may fail to meet the requirements listed.

9.5.5.6 For the opening CCV of the optional analysis of PAHs and PCP using the full scan method or the SIM technique, up to two target analytes and/or DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed in Table 5 - Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds. Up to two target analytes and/or DMCs (excluding those with maximum %D requirements of ±40.0%) may fail to meet the criteria listed. For a closing CCV, up to two target analytes and/or DMCs (excluding those with minimum RRF requirements of 0.010) may fail to meet the criteria listed.

9.5.5.7 Excluding those ions in the solvent front, no quantitation ion may saturate the detector. Consult the manufacturer's instrument operating manual to determine how saturation is indicated for your instrument.

9.5.6 Corrective Action for Continuing Calibration Verification

9.5.6.1 If the opening CCV technical acceptance criteria in Section 9.5.5 are not met, recalibrate the GC/MS instrument according to Section 9.3. Any samples or required blanks analyzed when opening CCV technical acceptance (including MS/MSDs) criteria have not been met will require reanalysis at no additional cost to the EPA. Refer to sample dilution procedure in Section 10.4.3 for target analytes that exceed the calibration range of the method.

9.5.6.2 If the closing CCV technical acceptance criteria in Section 9.5.5 are not met, then all associated samples and blanks analyzed within that 12-hour period must be reanalyzed at no additional cost to the EPA. The laboratory must carefully document the situations in the SDG Narrative. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the CCV technical acceptance criteria.

9.5.6.3 If this reanalysis is unsuccessful and the laboratory has evidence or suspects, because of preliminary results, sample color, or physical properties, that a sample or samples (including requested SIM analysis) may contain high concentrations of either target or non-target compounds that may be impacting the stability of the analytical system (i.e., causing closing CCV failures), then the Contractor shall proceed with the sample dilution/reanalysis procedure in Section 10.4.4.

9.5.6.4 The corrective action for sample reanalysis is not required when noncompliant analytes or associated DMCs, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or associated DMCs for which the dilution, re-extraction, or reanalysis was intended.

10.0 PROCEDURE

The Contractor must have the capability to perform all the sample cleanup procedures presented in this Exhibit. The Contractor may use any of the procedures or combinations of procedures to clean up the samples prior to analysis, unless the Contractor is specifically directed by the EPA Region to use a particular cleanup procedure or combination of cleanup procedures.

The Contractor must demonstrate that each cleanup procedure is capable of producing data that meets the technical acceptance criteria for the method, including MDLs (Section 12.4) and any precision and recovery limits.

NOTE: If SIM analysis of PAHs and PCP is requested for a sample, a full scan analysis at the regular concentration levels must be performed on that sample prior to the SIM analysis. For all SIM target analytes detected at or above CRQLs during the full scan analysis, a SIM analysis is not to be performed for those target analytes. Any SIM analyses not performed for this reason must be noted in the SDG Narrative.

10.1 Sample Preparation

10.1.1 Water and Leachate Samples

Continuous liquid-liquid extraction is used to extract the samples. Separatory funnel extraction or other manual extraction techniques cannot be used. Allow the sample to warm to ambient temperature before extraction.

10.1.1.1 Continuous Liquid-Liquid Extraction without Hydrophobic Membrane

10.1.1.1.1 Follow the manufacturer’s instructions for set-up.

10.1.1.1.2 Add 300-500 mL of methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom sidearm.

10.1.1.1.3 If the samples have been received in 1 L bottles, the Contractor shall mark the meniscus and transfer the entire sample into the continuous liquid-liquid extraction apparatus. If the sample was not received in a 1 L bottle, measure out a 1 L sample aliquot in a separate, clean graduated cylinder and transfer the aliquot to the continuous extractor.

10.1.1.1.4 Using a syringe or volumetric pipette, add 500 µL of the DMC spiking solution to result in the addition of 40 µg of each DMC and (if SIM is requested) 0.4 µg of the SIM DMCs (fluoranthene-d_{10} and 2-methylnaphthalene-d_{10})(Section 7.2.2.5) into the sample and mix well. Perform spiking prior to pH adjustment or any other processing steps.

10.1.1.1.5 Measure the pH of the sample with narrow range pH paper or a pH meter and record the pH. Adjust the pH to 2.0 with 1:1 sulfuric acid if required. Samples requiring pH adjustment must be noted in the SDG Narrative.

NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.1.1.6 Rinse the 1 L sample bottle and/or graduated cylinder with a small amount of methylene chloride and transfer the rinsate to the continuous extractor. Measure and record the volume of sample contained in the 1 L sample bottle with water, using a graduated cylinder.

10.1.1.1.7 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5-15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 18 hours.

NOTE 1: When a minimum drip rate of 10-15 mL/minute is maintained throughout the extraction, the extraction time may be reduced to a minimum of 12 hours. Allow to cool and then detach the distillation flask. Proceed to Section 10.2.

NOTE 2: Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.1.2 Continuous Liquid-Liquid Extraction with Hydrophobic Membrane

10.1.1.2.1 Follow the procedure in Sections 10.1.1.1 - 10.1.1.5, but reduce the amount of methylene chloride used to 50 mL and extract for a minimum of 6 hours.

10.1.1.2.2 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours.

10.1.1.2.3 Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion that will consume the solvent volume, preventing the efficient extraction of the sample. When this occurs, add additional solvent to ensure efficient extraction of the sample and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used. Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

10.1.1.2.4 Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

If low DMC recoveries occur, ensure that: 1) the apparatus was properly assembled to prevent leaks, 2) the drip rate/solvent cycling was optimized, and 3) there was proper cooling for condensation of solvent. Document the problem and the corrective action.

10.1.1.2.5 Alternate continuous extractor types that meet the requirements of the analytical method may also be used. If using alternate extractors or design types, follow the manufacturer's instruction for set-up. Optimize the extraction procedure.

10.1.2 Soil/Sediment Samples

Mix samples thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase.

10.1.2.1 Mandatory Determination of Concentration Level

10.1.2.1.1 The Contractor must determine whether a soil/sediment sample should be analyzed by the low-level or medium-level soil/sediment method. It is the responsibility of the Contractor to analyze the sample at the correct level.

10.1.2.1.2 When there is doubt as to the best approach, the Contractor should begin by processing the sample as low level. However, any subsequent unnecessary reanalysis at the medium level shall not be billable to the EPA.

10.1.2.1.3 Use of an EPA screening procedure or an in-house laboratory screening procedure is strongly encouraged. The procedure must be documented and available for review during on-site laboratory evaluation.

10.1.2.2 Low-Level Extraction of Soil/Sediment Samples

10.1.2.2.1 Three procedures are provided for the extraction of semivolatile analytes from low-level soil/sediment samples:

- ultrasonic extraction;
- Soxhlet extraction (automated and manual); and
- pressurized fluid extraction (PFE).

NOTE: All low-level soil/sediment samples in a Case must be extracted by the same procedure.

10.1.2.2.2 For soil/sediment sample extractions, perform the following steps rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample to the nearest 0.1 g, into a 400 mL beaker. If the system cannot accommodate 30 g of a sample, a smaller sample size may be used. The specified CRQLs must be met. Adjust the amount of solvents and standards added as necessary. Document the smaller sample size in the SDG Narrative along with all steps taken to ensure sample homogeneity.

10.1.2.2.3 Add 60 g of anhydrous powdered or granulated sodium sulfate, or 30 g of Hydromatrix™, and mix well to produce a sandy texture. Add additional drying agent as needed.

NOTE: For samples extracted by the PFE procedure (Section 10.1.2.2.7), the use of sodium sulfate is not recommended. As applicable, follow the manufacturer's instructions for use of all extraction equipment.

10.1.2.2.4 Add 500 µL of the DMC spiking solution to result in the addition of 40 µg of each DMC or if SIM is requested, 0.40 µg of the SIM DMCs (fluoranthene-d_{10} and 2-methylnaphthalene-d_{10})(Section 7.2.2.5.2) to the sample. Proceed to Section 10.1.2.2.5 for ultrasonic extraction, Section 10.1.2.2.6 for automated Soxhlet extraction, or Section 10.2.2.7 for pressurized fluid extraction.

10.1.2.2.5 Ultrasonic Extraction

10.1.2.2.5.1 Add 100 mL of 1:1 (v/v) acetone/methylene chloride.

10.1.2.2.5.2 Place the bottom of the tip of the 3/4-inch tapered disrupter horn about 1/2 inch below the surface of the solvent, but above the sediment layer. Do not use a microtip probe.

10.1.2.2.5.3 Sonicate for 3 minutes with output set at full power with pulse on (pulse energy as opposed to continuous) and percent duty cycle knob set at 50%.

NOTE: Refer to the manufacturer's instructions for appropriate output settings.

10.1.2.2.5.4 Transfer and filter extracts through Whatman No. 42 (or equivalent) filter paper using vacuum filtration or centrifuge and decant extraction solvent.

10.1.2.2.5.5 Repeat the extraction two more times with two additional 100 mL portions of 1:1 (v/v) acetone/methylene chloride. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with a clean spatula. Transfer the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 (v/v) acetone/methylene chloride.

10.1.2.2.5.6 If the sample is to be screened following the low-level preparation method prior to GPC, proceed to the appropriate screening procedure. Otherwise, proceed to Section 10.2.

10.1.2.2.6 [Automated] Soxhlet Extraction

The Contractor may use either automated or non-automated Soxhlet extraction. The following procedure is based on the use of a Soxtec HT-6 automated Soxhlet extraction system. When using a different system, refer to the instructions provided by the manufacturer for the appropriate procedure.

10.1.2.2.6.1 Check the heating oil level in the automated Soxhlet unit and add oil if needed. Follow the manufacturer's instructions to set the temperature on the service unit.

10.1.2.2.6.2 Press the "MAINS" button and observe that the switch lamp is now "ON". Open the cold water tap for the reflux condensers. Adjust the flow to 2 L/minute to prevent solvent loss through the condensers.

10.1.2.2.6.3 Transfer the entire sample from the beaker (Sections 10.1.2.2.2 - 10.1.2.2.4) to the thimble.

10.1.2.2.6.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

10.1.2.2.6.5 Insert the extraction cups containing boiling chips, and load each with appropriate volume of extraction solvent 1:1 (v/v) acetone/methylene chloride. Using the cup holder, lower the locking handle, ensuring that the safety catch engages. The cups are now clamped into position.

NOTE: The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.

10.1.2.2.6.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

10.1.2.2.6.7 Move the extraction knobs to the "RINSING" position. The thimbles will now hang above the solvent surface. Set the timer for 60 minutes. Condenser valves are still open. Extract for the preset time. After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise.

10.1.2.2.6.8 When all but 2-5 mL of the solvent have been collected, open the system and remove the cups. Transfer the contents of the cups to graduated, conical-bottom glass tubes. Rinse the cups with methylene chloride and add the rinsates to the glass tubes.

10.1.2.2.6.9 If the sample is to be screened following the low-level preparation method prior to GPC, proceed to the appropriate screening procedure. Otherwise, proceed to Section 10.2.

10.1.2.2.7 Pressurized Fluid Extraction

10.1.2.2.7.1 Transfer the entire sample from the beaker (Sections 10.1.2.2.2 - 10.1.2.2.4) to an extraction cell of the appropriate size for the aliquot.

10.1.2.2.7.2 Place the extraction cell into the instrument or autosampler tray, as described by the instrument manufacturer.

10.1.2.2.7.3 Place a pre-cleaned collection vessel in the instrument for each sample, as described by the instrument manufacturer. The total volume of the collected extract will depend on the specific instrumentation and the extraction procedure recommended by the manufacturer and may range from 0.5-1.4 times the volume of the extraction cell. Ensure that the collection vessel is sufficiently large to hold the extract.

10.1.2.2.7.4 The following are recommended extraction conditions:

Oven temperature	100°C
Pressure	1500-2000 psi
Static time	5 min. (after 5 min. pre-heat equilibration)
Flush volume	60% of the cell volume
Nitrogen purge	60 sec. at 150 psi (purge time may be extended for larger cells)
Static cycles	1

10.1.2.2.7.5 Optimize the extraction conditions as needed, according to the manufacturer’s instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500-2000 psi should suffice. An appropriate amount of 1:1 (v/v) acetone/methylene chloride should be used to achieve the conditions in Section 10.1.2.2.7.4.

10.1.2.2.7.6 Once established, the same pressure should be used for all samples in the same SDG.

10.1.2.2.7.7 Begin the extraction according to the manufacturer's instructions. Collect each extract in a clean vial. Allow the extracts to cool after the extractions are complete.

10.1.2.2.7.8 If the sample is to be screened following the low-level preparation method prior to GPC, proceed to the appropriate screening procedure. Otherwise, proceed to Section 10.2.

10.1.2.3 Medium-Level Extraction of Soil/Sediment Samples

The procedure described below is for the extraction of soil/sediment samples by the ultrasonic method (Section 10.1.2.2.5). The Contractor may also use the [automated or manual] Soxhlet extraction or PFE procedures described in Sections 10.1.2.2.6 and 10.1.2.2.7, respectively. The requirements of this analytical method must be met at all times (i.e., sample weight used for medium-level soil/sediment extraction and original CRQLs for medium-level soils). As applicable, follow the manufacturer's instructions for the use of all extraction equipment.

NOTE: All medium-level soil/sediment samples in a Case must be extracted by the same procedure.

10.1.2.3.1 Transfer approximately 1 g (record weight to the nearest 0.1 g) of sample to a 20 mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

10.1.2.3.2 Add 500 µL of DMC spiking solution to result in the addition of 40 µg of each DMC excluding the two SIM DMCs (fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}) (Section 7.2.2.5.2) to the sample mixture.

10.1.2.3.3 Add 2.0 g or sufficient quantity of anhydrous powdered or granulated sodium sulfate or Hydromatrix™ to the sample in the 20 mL vial and mix well to produce a sandy texture.

10.1.2.3.4 Immediately add sufficient methylene chloride to the sample so that the total volume is approximately 10 mL and disrupt the sample with the 1/8-inch tapered microtip ultrasonic probe for 2 minutes at output control setting 5, in continuous mode. Before extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. Decant and filter extract through Whatman No. 42 (or equivalent) filter paper using vacuum filtration or centrifuge and decant extraction solvent.

NOTE: Concentration of the extracts of soil/sediment samples prepared by the medium-level procedure described above may not be necessary. Proceed to Section 10.2.1.7 if no extract concentration is to be performed.

10.2 Extract Concentration

10.2.1 Concentration by Kuderna-Danish

10.2.1.1 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D, if equivalency is demonstrated for all the semivolatile

target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits.

10.2.1.2 For water samples, transfer the extract to a K-D concentrator by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate.

10.2.1.3 For soil/sediment samples, directly transfer the extract to the K-D concentrator, if the extract is known to be dry.

10.2.1.4 Rinse the original container collecting the extract (for both water and soil/sediment samples) and the column (for water samples) with at least two 20-30 mL portions of methylene chloride to complete the quantitative transfer.

10.2.1.5 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top of the column. Place the K-D apparatus in a hot water bath (60-70°C recommended) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-15 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 or 2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

10.2.1.6 For water extracts that do not require GPC cleanup, proceed to final concentration of extract (Section 10.2.2). Oily water samples extracts require GPC cleanup.

10.2.1.7 For water extracts that require GPC cleanup, adjust the volume of the extract to 10.0 mL with methylene chloride and proceed with GPC cleanup (Section 10.3).

10.2.1.8 For soil/sediment extracts, adjust the volume of the extract to 10.0 mL with methylene chloride and proceed with GPC cleanup (Section 10.3).

10.2.1.9 For water or soil/sediment extracts that have undergone GPC cleanup, proceed to final concentration of extract (Section 10.2.2).

10.2.2 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to volume before instrument analysis. They are the Micro Snyder Column and the Nitrogen Evaporation Technique.

10.2.2.1 Micro Snyder Column Technique

10.2.2.1.1 Add another one or two clean boiling chips to the concentrator tube and attach a two-ball Micro Snyder Column. Pre-wet the Snyder column by adding about 0.5 mL of methylene chloride to the top of the column. Place the K-D apparatus in a hot water bath (60-70°C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as

required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and lower joint into the concentrator tube with 0.20 mL (0.10 mL for low-level soil/sediment samples and water samples that have undergone GPC cleanup) of methylene chloride.

10.2.2.1.2 Adjust the final volume to 1.0 mL (0.50 mL for low-level soil/sediment samples and water samples that have undergone GPC cleanup) with methylene chloride. Transfer the extract to the PTFE-sealed screw-cap bottle, label the bottle, and store at ≤6°C. If no further cleanup is needed, proceed to Section 10.4 for GC/MS analysis.

10.2.2.2 Nitrogen Evaporation Technique

10.2.2.2.1 Place the concentrator tube in a warm water bath (30-35°C recommended) and evaporate the solvent volume to just below 1 mL (below 0.50 mL for low-level soil/sediment samples and water samples that have undergone GPC cleanup) using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). DO NOT ALLOW THE EXTRACT TO GO DRY.

10.2.2.2.2 Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or PTFE tubing. Plastic tubing must not be used between the carbon trap and the sample, as it may introduce interferences. The internal wall of the concentrator tube must be rinsed down several times with methylene chloride during the operation.

10.2.3 Final Extract Volumes

The final extract volumes in Sections 10.2.3.1 and 10.2.3.2 are recommended volumes. If more sensitive GC/MS systems are employed, then the larger extract volumes (less concentrated extracts) may be used, provided that the CRQLs for all target analytes can be achieved, and that all DMCs and internal standards have an expected extract concentration that is at the mid-point of the calibration curve.

10.2.3.1 Water

For water samples that did not undergo GPC cleanup, the extract must be brought to a final volume of 1.0 mL with methylene chloride. Remove boiling chips before adjusting final volume. For water samples that underwent GPC cleanup, the extract must be brought to a final volume equal to V_{out} (volume of extract collected from GPC cleanup) with methylene chloride [concentrating the extract to 0.50 mL will result in no loss of sensitivity despite the volume of extract (5.0 mL) not recovered after GPC cleanup].

10.2.3.2 Soil/Sediment

Adjust the final volume for low-level and medium-level soil/sediment samples to equal V_{out} with methylene chloride. For example, if V_{out} equals 0.50 mL, then the final volume must be adjusted to 0.50 mL. Concentrating the extract to 0.50 mL will result in no loss of sensitivity despite the volume of extract not recovered after GPC cleanup. Remove boiling chips before adjusting final volume.

10.2.3.3 Transfer the extract to a PTFE-sealed screw-cap bottle, label the bottle, and store at ≤6°C, but not frozen.

10.3 Cleanup by Gel Permeation Chromatography

10.3.1 Introduction

10.3.1.1 GPC is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the size of the molecules to be separated.

10.3.1.2 GPC must be performed for all soil/sediment extracts. GPC may be performed for water extracts that contain higher molecular weight contaminants that interfere with the analysis of the target analytes. In addition, GPC must be performed for all associated blanks and MS/MSDs. If the cleanup procedure is inadequate, contact the Sample Management Office (SMO).

10.3.2 GPC Column Preparation

Prepare the GPC column using Bio Beads. Alternative column packings may be used if: 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration verification, and 2) the column packings do not introduce contaminants/artifacts into the sample that interfere with the analysis of the semivolatile analytes. Follow the manufacturer’s instructions for preparation of the GPC column.

10.3.3 Calibration of GPC

10.3.3.1 Summary of GPC Calibration

The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column.

10.3.3.2 Frequency of GPC Calibration

Each GPC system must be calibrated prior to processing samples under the contract, when the GPC calibration verification solution fails to meet criteria (Section 10.3.3.4), when the column is changed, when channeling occurs, and once every 7 days when in use. Also, the retention time (RT) shift must be less than 5% when compared to RTs in the last calibration UV traces.

10.3.3.3 Procedure for GPC Calibration

Follow the manufacturer’s instructions for operating the GPC system. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte RTs and must be monitored.

10.3.3.3.1 Using a 10 mL syringe, load the calibration solution (Section 7.2.2.4) onto the GPC. Establish appropriate "COLLECT" and "DUMP" time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis(2-ethylhexyl)phthalate and after the elution of corn oil. Stop eluate collection shortly after the elution of perylene. Collection should be stopped before sulfur elutes. Use a "WASH" time of 10 minutes after the elution of sulfur. Each laboratory is required to establish its specific time sequences.

10.3.3.3.2 Reinject the calibration solution after appropriate "COLLECT" and "DUMP" cycles have been set, and the solvent flow and column pressure have been established.

10.3.3.3.3 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate problems with the system during sample processing.

10.3.3.3.4 Analyze a GPC blank of methylene chloride after each GPC calibration or each GPC calibration verification. Concentrate the methylene chloride that passes through the system during the "COLLECT" cycle using a K-D evaporator. Add internal standards at the appropriate concentration and analyze the concentrate by GC/MS.

10.3.3.4 Technical Acceptance Criteria for GPC Calibration

10.3.3.4.1 The GPC system must be calibrated at the frequency described in Section 10.3.3.2. The UV trace must meet the following requirements:

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution;
- Corn oil and the phthalate peaks should exhibit greater than 85% resolution;
- Phthalate and methoxychlor peaks should exhibit greater than 85% resolution;
- Methoxychlor and perylene peaks should exhibit greater than 85% resolution; and
- Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

10.3.3.4.2 The solvent flow rate and column pressure must be within the manufacturer’s specified ranges.

10.3.3.4.3 The RTs for bis(2-ethylhexyl)phthalate and perylene must not vary more than 5% between calibrations. Excessive RT shifts are caused by the following:

- Poor laboratory temperature control or system leaks;
- An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight; and/or
- Excessive laboratory temperatures causing outgassing of the methylene chloride.

10.3.3.4.4 The analyte concentrations in the GPC blank must be less than the CRQL for all target analytes in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits, except bis(2-ethylhexyl)phthalate, which must be less than 5 times the CRQL.

10.3.3.4.5 A copy of the two most recent UV traces of the calibration solution must be submitted with the data for the associated samples.

10.3.3.5 Corrective Action for GPC Calibration

10.3.3.5.1 If the requirements in Section 10.3.3.4 cannot be met, the column may be cleaned by processing several 5 mL volumes of butylchloride through the system. Butylchloride removes the discoloration and particles that may have precipitated out of the methylene chloride extracts. If a guard column is being used, replace it with a new one. This may correct the problem. If column maintenance does not restore the performance of the column, the column must be repacked with new packing and recalibrated. It may be necessary to obtain a new lot of Bio Beads if the column fails all criteria.

10.3.3.5.2 If the flow rate and/or column pressure do not fall within the manufacturer's specified ranges, a new column should be prepared.

10.3.3.5.3 A UV trace that does not meet the criteria in Section 10.3.3.4.1 would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

10.3.3.5.4 If the GPC blank exceeds the requirements in Section 10.3.3.4.4, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

10.3.4 GPC Calibration Verification

10.3.4.1 Summary of GPC Calibration Verification

The GPC calibration must be routinely verified with the calibration verification check mixture according to Exhibit D – Pesticides Analysis (Section 10.3.1.4).

10.3.4.2 Frequency of GPC Calibration Verification

10.3.4.2.1 The calibration verification must be performed at least once every 7 days (immediately following the GPC Calibration) whenever samples (including MS/MSDs and blanks) are cleaned up using the GPC.

10.3.4.2.2 Some samples may contaminate the SX-3 Bio Beads and change the retention volume of the GPC column. Therefore, system calibration and analyte recovery must be checked whenever a sample causes significant discoloration of the GPC column. Even if no darkening is visible, GPC calibration must be checked not less than once every 7 days.

10.3.4.3 Procedure for GPC Calibration Verification

The GPC calibration verification solution contains six pesticide target analytes that are not included in the calibration standards (Section 7.2.2.1); therefore, the Contractor must follow the GPC calibration verification procedure according to Exhibit D – Pesticides Analysis instructions (Section 10.3.1.4) prior to the analysis of semivolatile target analytes in samples, blanks, and MS/MSDs. The Contractor shall establish pesticide initial calibration prior to GPC calibration verification even if the samples are not scheduled for pesticide analysis.

10.3.4.3.1 The pesticide GPC calibration verification solution contains gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin.

10.3.4.3.2 Load the 5 mL sample loop by using a 10 mL syringe containing 8 mL of the pesticide GPC calibration verification solution. Fractions are collected in an autosequence by using the GPC program established by the UV detector calibration procedure (Section 10.3.3.3).

10.3.4.3.3 The collected GPC calibration verification fraction is transferred to a K-D apparatus, and the collection vessel is rinsed with two additional 10 mL portions of methylene chloride to complete the transfer. The volume of methylene chloride is reduced according to Section 10.2.1. After cooling, the solvent is exchanged to hexane according to the instructions in Exhibit D - Pesticide Analysis (Section 10.2.2). The final volume is adjusted to 10 mL, and the sample is analyzed by GC/Electron Capture Detector (ECD) according to the procedure in Exhibit D - Pesticides Analysis (Section 10.4). The analysis must be performed on only one of the GC/ECD columns used for pesticides analysis.

10.3.4.3.4 The recovery of each analyte must be determined for evaluation and reporting purposes. Calculate the Percent Recovery (%R) of each analyte using Equation 13 in Exhibit D - Pesticides Analysis (Section 10.3.2).

10.3.4.4 Technical Acceptance Criteria for GPC Calibration Verification

The technical criteria specified in Exhibit D – Pesticides Analysis must be met prior to the GPC cleanup on samples, blanks, and MS/MSDs.

10.3.4.5 Corrective Action for GPC Calibration Verification

The Contractor may continue to use the GPC column if the technical acceptance criteria for the GPC calibration verification are met. If the recoveries are outside of the acceptance criteria, the columns must be replaced and the GPC recalibrated according to the instructions in Section 10.3.3 and Section 10.3.4 before proceeding with any GPC cleanup on samples (including LCSs and MS/MSDs) and required method blanks.

10.3.5 Daily Ultraviolet Calibration Check (Optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (Section 7.2.2.4) and the UV detector calibration procedure (Section 10.3.3). The UV detector should be used to monitor the elution times for the phthalate, methoxychlor, and perylene, in that order. The precalibrated GPC program should "DUMP" greater than 85% of the phthalate and should "COLLECT" greater than 95% of the methoxychlor and perylene. Significant changes in elution times of the analytes (e.g., greater than 30 seconds) indicate that the column is out of calibration and must be recalibrated or replaced.

10.3.6 Sample Extract Cleanup by GPC

10.3.6.1 Summary of GPC Cleanup

10.3.6.1.1 It is very important to have constant laboratory temperatures during an entire GPC analysis, which could be 24 hours or more. If temperatures are not constant, RTs will shift, and the "DUMP" and "COLLECT" times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C.

10.3.6.1.2 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 (v/v) glycerol/water solution must be diluted and loaded into several loops. Similarly, extracts containing more than the manufacturer recommended non-volatile residue must be diluted and loaded into several loops. The non-volatile residue may be determined by evaporating a 100 µL aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

10.3.6.1.3 Systems using automated injection devices to load the sample on the column must be carefully monitored to assure that the required amount is being injected on the column. Viscous extracts or extracts containing a large amount of non-volatile residue will cause problems with injecting the proper amount of sample extract onto the column using automated injection systems. After the sample extract has been processed, the remaining sample extract in the injection vial must be checked to ensure the proper amount was injected on the column. If the proper amount of extract was not injected, the sample must be reprepared at no additional cost to the EPA, and the sample extract must either be diluted and loaded into several loops, or the sample extract must be injected manually.

10.3.6.2 Frequency of Sample Extract Cleanup by GPC

GPC cleanup must be performed once for each soil/sediment and all associated QC samples (blanks, LCSs, and MS/MSDs) must be subjected to this procedure. GPC cleanup on the method blank must be performed after all associated samples have been cleaned up (GPC sequence: calibration, sample 1, sample 2, etc., method blank, calibration verification).

10.3.6.3 Procedure for Sample Extract Cleanup by GPC

10.3.6.3.1 Particles greater than 5 microns may scratch the valve, which may result in a system leak and cross-contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Draw the sample extract through the filter assembly and into the 10 mL syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container (e.g., a 15 mL culture tube with a PTFE-lined screw-cap).

10.3.6.3.2 Alternatively, draw the extract into the syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 mL of extract into a 10 mL syringe.

NOTE 1: Some GPC instrument manufacturers recommend using a smaller micron size filter. Follow the manufacturer's recommended operating instructions.

NOTE 2: INTRODUCTION OF PARTICULATES OR GLASS WOOL INTO THE GPC SWITCHING VALVES MAY REQUIRE FACTORY REPAIR OF THE APPARATUS.

10.3.6.3.3 Follow the manufacturer's instructions for operation of the GPC system being utilized. A 2 mL injection loop may be used in place of a 5 mL injection loop. If a 2 mL injection loop is used, concentrate the sample extract to 4 mL instead of 10 mL, and then inject 4 mL instead of 10 mL.

10.3.6.3.4 If the sample is difficult to load, part of the system may be blocked. Take appropriate corrective action, following the manufacturer's recommendations. The problem must be resolved prior to loading sample extracts.

10.3.6.3.5 After loading each sample loop, wash the loading port with methylene chloride to minimize cross-contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

10.3.6.3.6 After loading the samples, process each sample using the "COLLECT" and "DUMP" cycle times established in Section 10.3.3.3.1.

10.3.6.3.7 Collect each sample in a 250 mL Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation, or directly into a K-D evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

- Change in solvent flow rate, caused by channeling in the column or changes in column pressure;
- Increase in column operating pressure due to the accumulation of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used; and/or
- Leaks in the system or significant variances in room temperature.

NOTE: Any samples that were loaded into multiple loops must be recombined before proceeding with concentration.

10.3.6.4 Final Concentration

Concentrate the extract as per Section 10.2.2. After removing boiling chips, final volumes should be brought to the volumes stated in Section 10.2.3.

10.4 Gas Chromatography/Mass Spectrometry Analysis

10.4.1 Introduction

Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, and CCV requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration. The same injection volume must be used for all standards, samples, and blanks.

10.4.2 Procedure for Sample Analysis by GC/MS

10.4.2.1 The internal standard spiking solution is added to an aliquot of each sample extract. Add sufficient amount of the internal standard spiking solution (Section 7.2.2.7) to each accurately measured aliquot of water, low-level, or medium-level soil/sediment sample extract to result in 20 ng/µL concentration of each internal standard.

NOTE: The internal standard spiking solution must be added to aliquots of sample extracts, not the entire extract, in order to make provision for sample dilutions and optional analysis of PAHs and PCP by the SIM technique, if requested.

10.4.2.2 If SIM is to be performed, the Contractor shall add sufficient amount of the internal standard spiking solution to each accurately measured aliquot of water and low-level soil/sediment sample extract to result in a 0.40 ng/µL concentration of each internal standard.

10.4.2.3 If sample extracts are to be diluted, add internal standards after dilution. Internal standards must be added to maintain the required 20 ng/µL (0.40 ng/µL for SIM) of each internal standard in the extract volume.

10.4.2.4 Inject 1.0 or 2.0 µL of the sample extract into the GC/MS.

10.4.3 Sample Dilutions

10.4.3.1 All samples must be analyzed undiluted.

10.4.3.2 If the concentration of any target analyte in any sample exceeds the concentration of the same target analyte in the high standard of the initial calibration, that sample extract must be diluted. Add the internal standard spiking solution to the diluted extract for a concentration of 20 ng/µL (0.40 ng/µL for optional analysis of PAHs and PCP by SIM) of each internal standard, and analyze the diluted extract. Guidance in performing dilution and exceptions to this requirement are given below.

10.4.3.3 Use the results of the original analysis to determine the approximate DF required for the analyte with the highest concentration to be within the initial calibration range.

10.4.3.4 The DF chosen must keep the concentration of the largest peak for a target analyte in the upper half of the calibration range of the instrument.

10.4.3.5 The maximum DF permitted for low-level soils is 30.0. If a low-level soil sample requires a DF greater than 30.0 to bring target analyte concentrations within the calibration range, then the medium-level method shall be utilized.

10.4.4 Procedure for Continually Failing Closing CCV

10.4.4.1 If the Contractor has followed the procedures in Sections 9.5.6 and 10.4.3, but the closing CCV is still not compliant with the criteria in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds, then the Contractor shall follow the procedures below.

10.4.4.1.1 Examine the sample data from the noncompliant sample sequence, including screening data if available, and segregate the samples that showed high levels of potential interference from those that appear normal.

10.4.4.1.2 The samples that appear not to contain significant interference (if any) shall be reanalyzed with appropriate dilutions in a new sequence or sequences that the closing CCV shall meet all technical acceptance criteria in Table 5 – Technical Acceptance Criteria for Initial Calibration, Initial Calibration Verification, and Continuing Calibration Verification for Semivolatile Organic Compounds.

10.4.4.1.3 Those samples in this segregated group that show high levels of interference but none of the detected target analytes would not require any further dilution per Section 10.4.3 above, shall be treated as follows: the steps in 10.4.4.1.3.1 – 10.4.4.1.3.3 shall be carried out only once for the affected samples.

10.4.4.1.3.1 Samples with a clearly defined baseline rise exceeding four times the peak height of the associated internal standards must be reanalyzed at a nominal 1:4 dilution (or further dilution), sufficient to reduce the baseline to within this factor of four criterion, including adjustment of the concentration of internal standard to that of a normal extract.

10.4.4.1.3.2 Samples that do not fit this description but still are suspected of containing significant interference shall be diluted 1:4, as described above.

10.4.4.1.3.3 These extracts shall be analyzed in a separate analytical sequence. The use of interstitial instrument blanks is required. If the closing CCV criteria are not met for this sequence, the Contractor shall document the procedure followed and any noncompliance in the SDG Narrative. All analyses of these samples shall be reported.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Analytes

11.1.1.1 The analytes listed in the TAL in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of the standard of the suspected analyte. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component within the GC RRT unit window established from the 12-hour calibration standard; and
- Correspondence of the sample component and calibration standard analyte mass spectra.

11.1.1.2 For establishing correspondence of the GC RRT, the sample component RRT must be within ±0.06 RRT units of the RRT of the corresponding continuing calibration standard component. For reference, the standard must be analyzed on the same 12-hour period as the sample. If samples are analyzed during the same 12-hour period as the initial calibration standards, use the RRT values from the 20 ng/µL standard (0.40 ng/µL for the calibration standard by SIM). Otherwise, use the corresponding opening CCV standard. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, then the RRT should be assigned by using EICPs for ions unique to the component of interest.

11.1.1.3 For comparison of standard and sample component mass spectra, mass spectra obtained from the Contractor's GC/MS (as opposed to library obtained spectra) are required. Once obtained, these standard spectra may be used for identification purposes, only if

the Contractor's GC/MS meets the daily instrument performance requirements for DFTPP. These standard spectra may be obtained from the standard analysis that was also used to obtain the RRTs.

11.1.1.4 The guidelines for qualitative verification by comparison of mass spectra are as follows:

11.1.1.4.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.1.1.4.2 The relative intensities of ions specified in the section above must agree within ±20% between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30-70%).

11.1.1.4.3 Ions greater than 10% in the sample spectrum, but not present in the standard spectrum, must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra.

11.1.1.4.4 If an analyte cannot be verified by all of the spectral identification criteria in Section 11.1.1.4, but in the technical judgment of the mass spectra interpretation specialist the identification is correct, then the Contractor shall report the identification and proceed with quantitation and document in the SDG Narrative.

11.1.2 Identification of Non-Target Compounds

11.1.2.1 A library search shall be executed for non-target compounds for the purpose of tentative identification. The NIST (2011 release or later), Wiley (2011 release or later), or equivalent mass spectral library shall be used as the reference library.

11.1.2.2 All organic compounds that have not been positively identified as semivolatile target analytes using the procedures detailed in Section 11.1.1, or that are not DMCs, internal standards, or volatile target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, unless the volatile analysis was not requested, shall be tentatively identified via a forward search of the NIST, Wiley, or equivalent mass spectral library. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer-generated library search routines must not use normalizations which would misrepresent the library or unknown spectra when compared to each other.

11.1.2.3 Up to 30 non-alkane Tentatively Identified Compounds (TICs) of greatest apparent concentration shall be reported on Form 1B-OR. Peaks that are tentatively identified as straight-chain, branched, or cyclic alkanes, and are alone or part of an alkane series, shall be reported as "total alkanes". An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The concentrations of each of the alkanes are to be summed and reported as a single result for the "total alkanes". The alkanes are not to be counted as part of the 30 compounds individually reported as TICs on Form 1B-OR. Carbon dioxide and compounds with responses less than 10% of the internal standard in which they are to be quantified (as determined by inspection of the peak areas or height) are not to

be reported (nor are they to be counted as part of the 30 compounds that are to be reported).

11.1.2.4 Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be searched, reported, and counted as part of the 30 most intense non-target semivolatile compounds, and qualified with an "A" flag on Form 1B-OR.

11.1.2.5 Rules for Making Tentative Identification

11.1.2.5.1 For compounds to be reported, as per the instructions in Section 11.1.2, identification (as generated by the library search program) of those receiving a library search match of 85% or higher should be considered a "probable match". The compound should be reported with the identification generated by the search program, unless the mass spectral interpretation specialist feels there is just evidence not to report the compound as identified by the library search program.

11.1.2.5.2 If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match. Do not report DMCs, internal standards, or analytes that are on the volatile or semivolatile TAL, unless the volatile analysis was not requested.

11.1.2.5.3 If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethylnaphthalenes), the compound with the highest library search percent match should be reported (or first compound if library search matches are the same).

11.1.2.5.4 If the mass spectral interpretation specialist has just evidence to support reporting a compound with a tentative identification of something other than that generated by the library search program (with a library search result of 85% or greater), the laboratory shall include in the SDG Narrative the justification for not reporting a compound as listed by the search program. This narrative shall detail explicitly why a library search generated identification for a compound was rejected. If a TIC has obvious isomer analogs, the laboratory shall include in the SDG Narrative a statement indicating that the exact isomer configuration, as reported, may not be absolutely accurate.

11.1.2.5.5 If the library search produces no matches at or above 85%, the mass spectral interpretation specialists are encouraged to make a valid tentative identification of the compound. If no valid tentative identification can be made, the compound should be reported as "unknown". The mass spectral interpretation specialist should give additional classification of the unknown, if possible (e.g., "unknown aromatic compound", "unknown chlorinated compound", etc.).

11.1.2.5.6 The Chemical Abstracts Service (CAS) registry number is the unique identifier for each chemical compound. As the rules of chemical nomenclature have changed over time, each chemical substance is liable to have several names or synonyms: trade or brand name(s); generic or common name(s); trivial or systematic; or International Union of Pure and Applied

Chemistry (IUPAC) name(s). Whether synonyms or other names are created for this compound, the CAS registry number will generally remain unchanged. The CAS registry number is simply an identifier which has no structural significance. Regardless of RTs, if the library search produces two or more compounds at or above 85% with the same Chemical Abstract Number, report the compound with the highest percent match (report first compound if the percent match is the same for two or more compounds) unless the mass spectral interpretation specialist feels there is just evidence not to report the compound with the highest match.

11.1.2.5.7 If the library search produces only one and the same compound (i.e., the same CAS registry number) with percent match at or above 85% at two different RTs, the compound having the highest percent match should be reported as TIC and the other one could be reported as unknown. If both TICs have the same percent match for the same compound, one of the TICs could be reported as unknown. Such justifications should be included in the SDG Narrative.

11.1.2.6 Qualitative identification of non-target compounds is not required when performing SIM analyses.

11.2 Quantitative Analysis

11.2.1 Data Processing Procedure

11.2.1.1 Target analytes identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (Table 9 – Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation and Table 10 – Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation of Polynuclear Aromatic Hydrocarbon and Pentachlorophenol). The EICP area of primary characteristic ions of analytes listed in Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards, are used for quantitation.

11.2.1.2 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitation. This normally occurs when there is compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific target analyte, DMC, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall also not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instance of manual integration must be documented in the SDG Narrative.

11.2.1.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS instrument 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

instrument operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s). This applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatile Target Analyte List and Contract Required Quantitation Limits, internal standards, and DMCs.

11.2.1.4 Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion, not when saturation occurs. If secondary ion quantitation is used, calculate an RRF using the area response (EICP) from the most intense secondary ion which is free of sample interferences, and document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

11.2.1.5 The factor $CV_{out}/(CV_{in} *E)$ used in Equations 7 to 10, will only apply when GPC is performed for semivolatile analysis. It is applied when GPC is performed (always for soil) to account for the factor of loss in the GPC, i.e., 50% efficiency, expressed as 0.50.

11.2.1.6 Target Analyte Calculations

Identified target analytes shall be quantitated by the internal standard method using Equation 7 or 8. The internal standard used shall be that which is assigned in Table 9 – Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation. The $\overline{RRF}$ from the initial calibration standard is used to calculate the concentration in the sample.

11.2.1.7 Water

EQ. 7 Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_x \times I_{is}}{A_{is} \times \overline{RRF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards)

A_{is} = Area of the characteristic ion (EICP) for the internal standard (Table 8 – Characteristic Ions for Semivolatile Target Analytes, Deuterated Monitoring Compounds, and Internal Standards). The target analytes are listed with their associated internal standards in Table 9 – Semivolatile Internal Standards with Associated Target and Deuterated Monitoring Compounds Assigned for Quantitation.

I_{is} = Amount of internal standard added, in ng

$\overline{RRF}$ = Mean Relative Response Factor determined from the initial calibration standard

V_i = Volume of extract injected, in µL

V_o = Volume of the water sample extracted, in mL

V_t = Volume of the concentrated extract, in µL

CV_{out} = Volume of extract produced by a cleanup process (cleanup and concentration), in µL

CV_{in} = Volume of extract subjected to a cleanup process, in µL

E = The efficiency of the cleanup process expressed as a fraction of material that passes through or is not mechanically lost during the cleanup step (e.g., 50% efficiency must be expressed as 0.50)

DF Dilution Factor. The DF for analysis of water samples for semivolatiles by this method is defined as follows:

$$DF = \frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated concentration by 1000.

11.2.1.8 Soil/Sediment

EQ. 8 Soil/Sediment Concentration

$$\text{Concentration}(\mu/kg) = \left(\frac{A_x \times I_{is}}{A_{is} \times \overline{RRF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

A_x, A_{is}, I_{is}, $\overline{RRF}$, DF, V_i, V_t, CV_{out}, CV_{in}, E = As given for water, as EQ. 7

W_t = Weight of the soil sample extracted, in g

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

11.2.2 Non-Target Compounds

11.2.2.1 An estimated concentration for TICs shall be determined by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used.

11.2.2.2 Equations 7 and 8 are also used for calculating TIC concentrations. Total area counts (or peak heights) from the total RICs are to be used for both the TIC to be measured (A_x) and the internal standard (A_{is}). An $\overline{RRF}$ of 1.0 is to be assumed.

11.2.3 Contract Required Quantitation Limit Calculations

11.2.3.1 Water

EQ. 9 Water and TCLP/SPLP Leachate Sample Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL})\left(\frac{V_x}{V_o}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

V_o, V_t, DF, CV_{out}, CV_{in}, E = As given in EQ. 7

Contract CRQL = The CRQL value reported in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contact Required Quantitation Limits

V_x = Method required sample volume (1000 mL)

V_y = Method required concentrated extract volume (1000 µL)

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated CRQL by 1000.

11.2.3.2 Soil/Sediment

EQ. 10 Soil/Sediment Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL}) \left(\frac{W_x}{W_t \times S}\right) \left(\frac{V_t}{V_y}\right) (\text{DF}) \left(\frac{CV_{out}}{CV_{in} \times E}\right)_1 \left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

W_t, V_t, DF, CV_{out}, CV_{in}, E = As given in EQ. 8

Contract CRQL = The CRQL value reported in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contact Required Quantitation Limits

W_x = Method required sample weight (30 g for low-level soil/sediment samples and 1.0 g for medium-level soil/sediment samples)

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

V_y = Method required concentrated extract volume (1000 µL)

11.2.4 Deuterated Monitoring Compound Recoveries

11.2.4.1 Calculate the concentration of each DMC using the same equation as used for the target analytes.

11.2.4.2 Calculate the recovery of each DMC in all samples and blanks using Equation 11. Report the recoveries on the appropriate forms.

EQ. 11 DMC Percent Recovery

$$\%\ R = \frac{Q_d \times DF}{Q_a} \times 100$$

WHERE,

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

DF = Dilution Factor

11.3 Technical Acceptance Criteria for Sample Analysis

11.3.1 The samples must be analyzed on a GC/MS system meeting the DFTPP, initial calibration, ICV, CCV, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the technical acceptance criteria for GPC calibration.

11.3.2 The sample must be extracted and analyzed within the contract holding times.

11.3.3 The sample must have an associated method blank meeting the method blank technical acceptance criteria.

11.3.4 The %R of each of the DMCs in a sample must be within the recovery limits listed in Table 11 – Deuterated Monitoring Compound Recovery Limits. Up to four DMCs per sample may fail to meet the recovery limits listed, but all %Rs must be greater than zero. The %R for 4-Chloroaniline-d_4 is advisory only. If the optional analysis of PAHs and PCP only by the full scan method is to be performed, no more than two DMCs per sample may fail to meet the recovery limits listed in Table 11 – Deuterated Monitoring Compound Recovery Limits, but all %Rs must be greater than zero. If the optional analysis of PAHs and PCP using the SIM technique is to be performed, both SIM DMCs must meet the recovery limits in Table 11 – Deuterated Monitoring Compound Recovery Limits. For TCLP leachate sample analysis, up to one DMC associated to the TCLP analytes may fail to meet the recovery limits listed in Table 11 – Deuterated Monitoring Compound Recovery Limits, but the %R must be greater than zero.

NOTE: The DMC recovery requirements do not apply to samples that have been diluted.

11.3.5 The EICP area for each of the internal standards in the sample must be within the range of 50.0%-200% of its response in the most recent opening CCV standard analysis.

11.3.6 The RT shift for each of the internal standards in the sample must be within ±30 seconds of its RT in the most recent opening CCV standard analysis.

11.3.7 Excluding those ions in the solvent front, no ion may saturate the detector. No target analyte concentration may exceed the upper limit of the initial calibration range unless a more diluted aliquot of the sample extract is also analyzed according to the procedures in Section 10.4.3.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or any samples not meeting the sample technical acceptance criteria will require re-extraction and/or reanalysis at no additional cost to the EPA.

11.4.2 Corrective actions for failure to meet technical acceptance criteria for instrument performance checks, initial calibration, ICV, and CCV must be completed before the analysis of samples.

11.4.3 If the technical acceptance criteria for any of the internal standards and DMCs are not met, check calculations, internal standard and DMC spiking solutions, and instrument performance. It may be necessary to bake out the system, to recalibrate the

instrument, or take other corrective action procedures to meet the technical acceptance criteria.

11.4.4 After completing the corrective actions outlined above, the Contractor shall proceed to the following corrective actions including: reanalyze the extract used for the initial analysis (i.e., reinject the same extract containing the internal standards); analyze a new aliquot of the original sample extract with freshly added internal standards; or re-extract and reanalyze the sample, as appropriate.

11.4.4.1 If the DMC recoveries do not meet the acceptance criteria in the initial sample extract analysis, re-extract the sample and analyze the extract.

- If the DMC recoveries meet the acceptance criteria in the re-extracted sample, it indicates that the problem was within the Contractor's control. Therefore, only submit the data from the reanalysis.
- If the DMC recoveries fail to meet the acceptance criteria in the re-extracted sample, then submit the data from both analyses. Distinguish between the initial analysis and the re-extracted analysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.2 If the internal standard compound responses do not meet the acceptance criteria in the initial sample extract analysis, reanalyze the extract used for the initial analysis (i.e., reinject the same extract containing the internal standards), or, in lieu of the reinjection, directly analyze a new aliquot of the original sample extract with freshly added internal standards.

- If the internal standard compound responses meet the acceptance criteria in the reanalyzed sample (reinjection or the reanalysis of the original sample extract with freshly added internal standards), it indicates that the problem was within the Contractor's control. Therefore, only submit the data from the reanalysis.
- If the internal standard compound responses are still noncompliant after the analysis of the extract with freshly added internal standards, the Contractor shall dilute the original sample extract by a factor of 2-10 and reanalyze the extract. If the internal standard compound responses are acceptable in any of the subsequent diluted analyses, submit both data from both the reanalysis and the compliant diluted analysis.
- If the internal standard compound responses fail to meet the acceptance criteria in the analysis of the extract with freshly added internal standards and the subsequent diluted analysis, submit the data from both analysis. Distinguish between the initial analysis and the re-extracted analysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.4.3 If both the DMC recoveries or internal standard compound responses do not meet the acceptance criteria in the initial sample extract analysis, re-extract the sample and analyze the new extract.

- If both the DMC recoveries and the internal standard compound responses meet the acceptance criteria in the re-extracted sample, it indicates that the problem was within the Contractor's control. Therefore, only submit the data from the reanalysis.
- If the DMC recoveries fail to meet the acceptance criteria in the re-extracted sample, then submit the data from both analyses. Distinguish between the initial analysis and the re-extraction/reanalysis in all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.
- If the internal standard compound responses fail to meet the acceptance criteria in the re-extracted sample, follow the corrective actions in Section 11.4.4.2 to perform reinjection of the re-extracted sample or reanalysis of the new aliquot of the re-extracted sample with the freshly added internal standards and the subsequent corrective actions if necessary; and submit the specified data.

11.4.4.4 If the DMC recoveries or internal standard compound responses in a sample used for the MS/MSD analyses are outside the acceptance criteria, the Contractor shall proceed to the following corrective actions:

- If the DMC recoveries in a sample used for the MS/MSD analyses are outside the acceptance criteria, then the sample shall be re-extracted/reanalyzed only if the DMC recoveries meet the acceptance criteria in both the MS and MSD analyses.
- If the internal standard compound responses do not meet the acceptance criteria, the Contractor shall proceed to the reanalysis in Sections 11.4.4.2 and 11.4.4.3 even if the internal standard compound responses meet the technical acceptance criteria in the MS/MSD analyses.

11.4.5 Corrective Action for Internal Standard Compound Retention Times Outside Acceptance Criteria

11.4.5.1 If the internal standard compound RTs are not within their acceptance criteria, check the instrument for malfunctions. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

11.4.5.2 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was matrix effect, take the corrective action steps:

- Reanalyze the sample extract. EXCEPTION: If the internal standard compound RTs in a sample used for an MS or MSD were outside the acceptance criteria, then it should be reanalyzed only if the internal standard compound RTs were within the acceptance criteria in both of the MS/MSD analyses.
- If the internal standard compound RTs are within the acceptance criteria in the reanalyzed sample extract, then the problem was within the Contractor's control. Therefore, submit only data from the reanalysis when the internal standard compound RTs within the acceptance limits.

- If the internal standard compound RTs are outside the acceptance criteria in the reanalyzed sample extract, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables, using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.6 If the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume, the Contractor shall contact SMO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Summary

There is one type of blank required by this method: the method blank.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate or Hydromatrix™ for soil/sediment samples) carried through the entire analytical procedure. The volume or weight or the reference matrix must be approximately equal to the volume of weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples. The leachate extraction blank shall be extracted and reported as SLEB## on Form 1A-OR and Form 1B-OR.

12.1.2.2 Frequency of Method Blank

A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples [excluding MS/MSDs and Performance Evaluation (PE) samples]. In addition, a method blank shall:

- Be extracted by the same procedure used to extract samples; and
- Be analyzed on each GC/MS system under the same conditions used to analyze associated.

12.1.2.3 Procedure for Method Blank

12.1.2.3.1 For water samples, measure 1.0 L volume of reagent water and spike with 40 µg of each DMC and, if SIM is requested, 0.40 µg of each SIM DMC (Section 7.2.2.5). For soil/sediment samples, measure 1 g (medium-level) or 30 g (low-level) of sodium sulfate or Hydromatrix™ and spike with 40 µg of each DMC and 0.40 µg (low-level) of each SIM DMC. Extract, concentrate, cleanup, and analyze the blank according to Section 10.0.

12.1.2.3.2 Under no circumstances should method blanks be analyzed at a dilution.

12.1.2.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.0.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 All blanks must be prepared and analyzed on a GC/MS system meeting the DFTPP, initial calibration, ICV, and CCV technical acceptance criteria and at the frequency described in Section 12.1.2.2.

12.1.2.5.2 The %R of each of the DMCs in the blank must be within the acceptance limits listed in Table 11 – Deuterated Monitoring Compound Recovery Limits. These limits are not advisory except for 4-Chloroaniline-d_4.

12.1.2.5.3 The blank must meet the sample acceptance criteria listed in Sections 11.3.4 - 11.3.7.

12.1.2.5.4 A method blank for semivolatile analysis for low-level soil and water samples must contain less than five times the CRQL of the bis(2-ethylhexyl) phthalate listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits. For all other target analytes, the method blank must contain less than the CRQL of any single target analyte (Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits). For medium-level soils, the method blank must contain less than the CRQL of any single target analyte.

12.1.2.5.5 All method blanks must be analyzed undiluted.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria, the Contractor shall consider the analytical system to be out of control.

12.1.2.6.2 If contamination is the problem, then the source of the contamination MUST be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples associated with a method blank that does not meet the method blank technical acceptance criteria will require re-extraction and reanalysis at no additional cost to the EPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvent, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the GC/MS be eliminated.

12.1.2.6.3 If DMC recoveries in the method blank do not meet the acceptance criteria listed in Section 12.1.2.5.2 and Table 11 - Deuterated Monitoring Compound Recovery Limits, first reanalyze the method blank. If the DMC recoveries do not meet the acceptance criteria after reanalysis, the method blank and all samples associated with that method blank must be re-extracted and reanalyzed at no additional cost to the EPA.

12.1.2.6.4 If the method blank does not meet internal standard response requirements listed in Section 11.3.5, follow the corrective action procedure outlined in Section 11.4.5. The Contractor shall resolve and document the resolution of the problem before proceeding with sample analysis.

12.1.2.6.5 If the method blank does not meet the RT requirements for internal standards (Section 11.3.6), check the instrument for malfunction and recalibrate. Reanalyze the method blank.

12.2 Matrix Spike and Matrix Spike Duplicate

12.2.1 Summary of Matrix Spike and Matrix Spike Duplicate

To evaluate the effects of the sample matrix on the methods used for semivolatile analyses, the EPA has prescribed a mixture of semivolatile target analytes to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method. An MS/MSD shall be extracted and analyzed only if requested by the EPA Region (through SMO) or specified on the Traffic Report/Chain of Custody (TR/COC) Record.

12.2.2 Frequency of Matrix Spike and Matrix Spike Duplicate

12.2.2.1 If requested, an MS/MSD must be performed for each group of 20 field samples of a similar matrix in an SDG. An MS/MSD should be analyzed for each sample matrix (water/soil) and each level (low/med). For the optional analysis by the SIM method, MS/MSD will not be required unless specifically requested by the EPA Region.

12.2.2.2 The Contractor shall not perform MS/MSD analysis on any of the field QC or PE samples.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample remaining to perform an MS/MSD, then the Contractor shall choose another sample on which to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify SMO that insufficient sample was received and identify the EPA sample selected for the MS/MSD analysis. SMO shall contact the EPA Region for confirmation immediately after notification. The rationale for the choice of another sample other than the one designated by the EPA shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform the requested MS/MSD, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the EPA Region for instructions. The EPA Region will either approve that no MS/MSD be performed, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the EPA Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2.1, the Contractor shall contact SMO. SMO will contact the EPA Region to determine which samples should have an MS/MSD analysis performed on them. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.6 When a Contractor receives only PE sample(s), no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD analysis when the EPA Region did not designate samples to be used for this purpose. If the PE sample is an ampulated standard, the ampulated PE sample is not considered to be another matrix type.

12.2.3 Procedure for Preparing Matrix Spike and Matrix Spike Duplicate

12.2.3.1 For water samples, prepare two additional 1 L aliquots of the sample chosen for spiking in two continuous extractors. Add 500 µL MS/MSD spiking solution corresponding to 40 µg of each DMC (0.40 µg of each SIM DMC if SIM is requested) and 500 µL MS/MSD spiking solution corresponding to 40 µg each of the Matrix Spike compound (0.40 µg of each Matrix Spike compound for SIM analysis). These additions shall be made to the samples prior to transferring to the continuous liquid-liquid extraction apparatus. Extract, concentrate, cleanup, and analyze the MS/MSD according to the procedures for water samples (Section 10.1.1).

NOTE: For analysis of PAHs and PCP only, add 500 µL of each DMC standard corresponding to 40 µg of each PAH and PCP (0.40 µg of each SIM DMC) and 500 µL MS/MSD standard to the corresponding to 40 µg each of acenaphthene, pyrene, and PCP Matrix Spike analytes (0.40 µg each of acenaphthene, pyrene, and PCP Matrix Spike compound for SIM analysis).

12.2.3.2 For low-level soil/sediment samples, prepare two additional 30 g aliquots (record weight to nearest 0.1 g) of the sample chosen for spiking in the two 400 mL beakers. Add 500 µL of the DMC spiking solution and 500 µL of the matrix spiking solution to each aliquot, to result in the addition of 40 µg of each DMC (0.40 µg of each SIM DMC) and 500 µL MS/MSD spiking solution corresponding to 40 µg of each Matrix Spike analyte (0.40 µg of each Matrix Spike analyte for SIM analysis). Add 60 g of anhydrous powdered sodium sulfate or 30 g of Hydromatrix™ to each aliquot. Mix well. Follow the appropriate extraction procedure in Section 10.1.2, extract, concentrate, cleanup, and analyze the MS/MSD according to the procedures for low-level soil samples.

NOTE: For analysis of PAHs and pentachlorophenol only, add 500 µL each DMC standard corresponding to 40 µg of each PAH and PCP (0.40 µg of each SIM DMC) and 500 µL MS/MSD spiking solution corresponding to 40 µg each of acenaphthene, pyrene, and PCP Matrix Spike compounds (0.40 µg each of acenaphthene, pyrene, and PCP Matrix Spike compound for SIM analysis).

12.2.3.3 For medium-level soil/sediment samples, prepare two additional 1.0 g aliquots (record weight to nearest 0.1 g) of the sample chosen for spiking in two 20 mL vials. Add a sufficient amount of DMC spiking solution and the matrix spiking solution to result in the addition of 40 µg of each DMC and 40 µg of each Matrix Spike analyte. Add 2.0 g of anhydrous powdered sodium sulfate or 1.0 g of Hydromatrix™ to each aliquot. Mix well. Proceed with the appropriate extraction procedure (Section 10.1.2.3). Extract, concentrate, cleanup, and analyze the MS/MSD according to the procedures for medium-level samples.

12.2.3.4 Before any MS/MSD analysis, analyze the original sample, then analyze the MS/MSD at the same concentration as the most concentrated extract for which the original sample results will be reported. For example, if the original sample is to be reported at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do not dilute the MS/MSD samples further to get either spiked or non-spiked analytes within calibration range.

Sample dilutions must be performed in accordance with Section 10.4.3.

NOTE: In cases where PAHs and PCP-only SIM MS/MSD is requested, and the sample designated for MS/MSD analysis has PAH target analytes and PCP detected at or above the sample adjusted CRQL or any target exceeding the calibration range, during the full scan analysis, then the laboratory must contact SMO to determine if another sample should be chosen for PAH and PCP only SIM MS/MSD analysis.

12.2.4 Calculations for Matrix Spike and Matrix Spike Duplicate

12.2.4.1 Calculate the concentrations of the Matrix Spike analytes using the same equations as used for target analytes (Equations 7 and 8). Calculate the recovery of each Matrix Spike analyte using the following equation:

EQ. 12 Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spike Sample Result

SR = Original Sample Result

SA = Spike Added

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each analyte in the MS/MSD using the following equation:

EQ. 13 Relative Percent Difference

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

WHERE,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

NOTE: The vertical bars in the equation above indicate the absolute value of the difference.

12.2.5 Technical Acceptance Criteria for Matrix Spike and Matrix Spike Duplicate

12.2.5.1 All MS/MSDs must be analyzed on a GC/MS system meeting DFTPP, initial calibration, ICV, CCV, and method blank technical acceptance criteria and at the frequency described in Section 12.2.2. The MS/MSD must undergo cleanup procedures when required on a GPC meeting the technical acceptance criteria for GPC calibration.

12.2.5.2 The MS/MSD must be extracted and analyzed within the contract holding time.

12.2.5.3 The RT shift for each of the internal standards must be within 30 seconds of its RT and the most recent opening CCV standard analysis.

12.2.5.4 The limits for MS analyte recovery and RPD are given in Table 12 - Matrix Spike Recovery and Relative Percent Difference Limits. As these limits are only advisory, no further action by the Contractor is required. However, frequent failure to meet the limits for recovery or RPD warrant investigation by the Contractor, and may result in questions from the EPA.

12.2.6 Corrective Action for Matrix Spike and Matrix Spike Duplicate

Any MS/MSD that fails to meet the technical acceptance criteria in Sections 12.2.5.1 and 12.2.5.3 must be reanalyzed at no additional cost to the EPA.

12.3 Laboratory Control Sample

Not applicable to this method.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed under the contract, the MDL for each semivolatile target analyte shall be determined on each instrument used for analysis. MDL determination is matrix-specific and level-specific (i.e., the MDL shall be determined for water, low-level soil/sediment, and medium-level soil/sediment samples). The MDLs must be determined annually thereafter and after major instrument maintenance. Major instrument maintenance includes, but is not limited to: cleaning or replacement of the mass spectrometer source, mass filters (e.g., quadrupole, ion trap, etc.), or electron multiplier (or similar device). A new MDL study will not be required after changing the GC column, as long as the replacement has the same length, inner diameter, and stationary phase.

12.4.2 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.3 The determined concentration of the MDL must be less than the CRQL listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and submitted to the EPA within seven (7) days of study completion. This schedule and the designated recipients are specified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 of Exhibit D – Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 of Exhibit D – Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Automated Soxhlet Extraction, SW-846 Method 3541, Revision 0, September 1994.

16.2 U.S. Environmental Protection Agency, Continuous Liquid-Liquid Extraction, SW-846 Method 3520C, Revision 3, December 1996.

16.3 U.S. Environmental Protection Agency, Pressurized Fluid Extraction (PFE), SW-846 Method 3545A, Revision 1, February 2007.

16.4 U.S. Environmental Protection Agency, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8270D, Revision 5, July 2014.

16.5 U.S. Environmental Protection Agency, Silica Gel Cleanup, SW-846 Method 3630C, Revision 3, December 1996.

16.6 U.S. Environmental Protection Agency, Ultrasonic Extraction, SW-846 Method 3550C, Revision 3, February 2007.

16.7 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS

Systematic Name	EPA Registry Name	Synonym	CAS #
1,4-Dioxane	1,4-Diethyleneoxide	Diethylene dioxide	123-91-1
Benzaldehyde	Benzaldehyde	Benzoic aldehyde	100-52-7
Phenol	Phenol	Hydroxybenzene	108-95-2
Ethane, 1,1'-oxybis[2-chloro-	Bis(2-chloroethyl)ether	Dichloroethyl ether	111-44-4
Phenol, 2-chloro-	o-Chlorophenol	2-Hydroxychlorobenzene	95-57-8
Phenol, 2-methyl-	o-Cresol	1-Hydroxy-2-methylbenzene	95-48-7
Phenol, 3-methyl-	m-Cresol	1-Methyl-3-hydroxybenzene	108-39-4
Propane, 2,2'-oxybis[1-chloro-	Bis(2-chloro-1-methylethyl)ether	1,1'-Dichlorodiisopropyl ether	108-60-1
Ethanone, 1-phenyl-	Acetophenone	Acetylbenzene	98-86-2
Phenol, 4-methyl-	p-Cresol	1-methyl-4-hydroxybenzene	106-44-5
1-Propanamine, N-nitroso-N-propyl-	N-Nitrosodi-n-propylamine	Di-n-propylnitrosamine	621-64-7
Ethane, 1,1,1,2,2,2-hexachloro-	Hexachloroethane	Carbon hexachloride	67-72-1
Benzene, nitro-	Nitrobenzene	Nitrobenzol	98-95-3
2-Cyclohexen-1-one, 3,5,5-trimethyl-	Isophorone	Isoacetophorone	78-59-1
Phenol, 2-nitro-	o-Nitrophenol	o-Hydroxynitrobenzene	88-75-5
Phenol, 2,4-dimethyl-	2,4-Dimethylphenol	1-Hydroxy-2,4-dimethylbenzene	105-67-9
Ethane, 1,1'-[methylenebis(oxy)]bis[2-chloro-	Bis(2-chloroethoxy)methane	Formaldehyde bis(2-chloroethyl) acetal	111-91-1
Phenol, 2,4-dichloro-	2,4-Dichlorophenol	1-Hydroxy-2,4-dichlorobenzene	120-83-2
Naphthalene	Naphthalene	Naphthalin	91-20-3
Benzenamine, 4-chloro-	4-Chloroaniline	4-Chloroaniline	106-47-8
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	Hexachlorobutadiene	Hexachloro-1,3-Butadiene	87-68-3
2H-Azepin-2-one, hexahydro-	Caprolactam	2-oxohexamethyleneimine	105-60-2
Phenol, 4-chloro-3-methyl-	p-Chloro-m-cresol	2-Chloro-5-hydroxytoluene	59-50-7
Naphthalene, 2-methyl-	2-Methylnaphthalene	β-Methylnaphthalene	91-57-6

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro-	Hexachlorocyclopentadiene	Hexachloro-1,3-cyclopentadiene	77-47-4
Phenol, 2,4,6-trichloro-	2,4,6-Trichlorophenol	Trichloro-2-hydroxybenzene	88-06-2
Phenol, 2,4,5-trichloro-	2,4,5-Trichlorophenol	Collunosol	95-95-4
1,1'-Biphenyl	Biphenyl	Phenylbenzene	92-52-4
Naphthalene, 2-chloro-	2-Chloronaphthalene	beta-Chloronaphthalene	91-58-7
Benzenamine, 2-nitro-	o-Nitroaniline	2-Nitroaniline	88-74-4
1,2-Benzenedicarboxylic acid, 1,2-dimethyl ester	Dimethyl phthalate	Phthalic acid, dimethyl ester	131-11-3
Benzene, 2-methyl-1,3-dinitro-	2,6-Dinitrotoluene	1-Methyl-2,6-dinitrobenzene	606-20-2
Acenaphthylene	Acenaphthylene	Cyclopenta[de]naphthalene	208-96-8
Benzenamine, 3-nitro-	m-Nitroaniline	3-Nitroaniline	99-09-2
Acenaphthylene, 1,2-dihydro-	Acenaphthene	1,8-Ethylenenaphthalene	83-32-9
Phenol, 2,4-dinitro-	2,4-Dinitrophenol	1-Hydroxy-2,4-dinitrobenzene	51-28-5
Phenol, 4-nitro-	p-Nitrophenol	p-Hydroxynitrobenzene	100-02-7
Dibenzofuran	Dibenzofuran	2,2'-Biphenylene Oxide	132-64-9
Benzene, 1-methyl-2,4-dinitro-	2,4-Dinitrotoluene	4-Methyl-1,3-Dinitrobenzene	121-14-2
1,2-Benzenedicarboxylic acid,1,2-diethyl ester	Diethyl phthalate	Phthalic acid, diethyl ester	84-66-2
9H-Fluorene	Fluorene	o-Biphenylenemethane	86-73-7
Benzene, 1-chloro-4-phenoxy-	p-Chlorophenylphenyl ether	4-Chlorophenylphenyl ether	7005-72-3
Benzenamine, 4-nitro-	p-Nitroaniline	4-Nitroaniline	100-01-6
Phenol, 2-methyl-4,6-dinitro-	4,6-Dinitro-o-cresol	4,6-Dinitro-2-methylphenol	534-52-1
Benzenamine, N-nitroso-N-phenyl-	N-Nitrosodiphenylamine	Diphenylnitrosamine	86-30-6
Benzene, 1,2,4,5-tetrachloro-	1,2,4,5-Tetrachlorobenzene	s-Tetrachlorobenzene	95-94-3
Benzene, 1-bromo-4-phenoxy-	p-Bromophenyl phenyl ether	4-Bromophenyl phenyl ether	101-55-3
Benzene, hexachloro-	Hexachlorobenzene	Hexachlorobenzol	118-74-1

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
1,3,5-Triazine-2,4-diamine, 6-chloro-N-ethyl-N'-(1-methylethyl)-	Atrazine	Fenatrol	1912-24-9
Phenol, 2,3,4,5,6-pentachloro-	Pentachlorophenol	Phenol, pentachloro	87-86-5
Phenanthrene	Phenanthrene	Phenanthrin	85-01-8
Anthracene	Anthracene	Paranaphthalene	120-12-7
9H-Carbazole	Carbazole	Diphenylenimine	86-74-8
1,2-Benzenedicarboxylic acid, dibutyl ester	Dibutyl phthalate	Di-n-butylphthalate	84-74-2
Fluoranthene	Fluoranthene	Benzo[j,k]fluorene	206-44-0
Pyrene	Pyrene	Benzo[d,e,f]phenanthrene	129-00-0
1,2-Benzenedicarboxylic acid, 1-butyl 2-(phenylmethyl) ester	Butyl benzyl phthalate	Phthalic acid, benzyl butyl ester	85-68-7
[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro-	3,3'-Dichlorobenzidine	o,o'-Dichlorobenzidine	91-94-1
Benz[a]anthracene	Benz[a]anthracene	1,2-Benzanthracene	56-55-3
Chrysene	Chrysene	1,2-Benzphenanthrene	218-01-9
1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester	Di(2-ethylhexyl) phthalate	phthalic acid, (2-ethyl hexyl)ester	117-81-7
1,2-Benzenedicarboxylic acid, 1,2-dioctyl ester	Di-n-octyl phthalate	n-Octyl phthalate	117-84-0
Benz[e]acephenanthrylene	Benzo(b)fluoranthene	2,3-Benzofluoranthene	205-99-2
Benzo[k]fluoranthene	Benzo[k]fluoranthene	11,12-Benzofluoranthene	207-08-9
Benzo[a]pyrene	Benzo[a]pyrene	3,4-Benzopyrene	50-32-8
Indeno[1,2,3-cd]pyrene	Indeno[1,2,3-cd]pyrene	1,10-(1,2-Phenylene)pyrene	193-39-5
Dibenzo[a,h]-anthracene	Dibenzo[a,h]-anthracene	1,2,5,6-Dibenzanthracene	53-70-3
Benzo[ghi]perylene	Benzo[ghi]perylene	1,12-Benzoperylene	191-24-2
Phenol,2,3,4,6-tetrachloro	2,3,4,6-Tetrachlorophenol	1-Hydroxy-2,3,4,6-tetrachlorobenzene	58-90-2

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
Internal Standards			
Benzene-d4, 1,4-dichloro-	1,4-Dichlorobenzene-d4	1,4-Dichloro-2,3,5,6-	3855-82-1
Naphthalene-d8	Naphthalene-d8	Tetradeuterobenzene	1146-65-2
Acenaphthylene-d8, 1,2-dihydro-d2-	Acenaphthene-d10	Perdeuteronaphthalene	15067-26-2
Phenanthrene-d10	Phenanthrene-d10	Phenanthrene, perdeutero-	1517-22-2
Chrysene-d12	Chrysene-d12	Chrysene, perdeutero-	1719-03-5
Perylene-d12	Perylene-d12	Perylene- perdeutero-	1520-96-3
DMCs			
1,4-Dioxane-2,2,3,3,5,5,6,6-d8	1,4-Dioxane-d8	1,4-Diethyleneoxide-d8	17647-74-4
Phen-d5-ol	Phenol-d5	Phenol-d5	4165-62-2
Ethane-1,1,2,2-d4, 1,1'-oxybis[2-chloro-	Bis(2-chloroethyl)ether-d8	Bis(2-chloroethyl)ether-d8	93952-02-4
Phen-2,3,4,5-d4-ol, 6-chloro-		2-chlorophenol-d4	93951-73-6
Phen-2,3,5,6-d4-ol-d, 4-(methyl-d3)-	4-methylphenol-d8	4-methylphenol-d8	190780-66-6
Benzen-2,3,5,6-d4-amine, 4-chloro	4-Chloroaniline-d4	4-Chloroaniline-d4	191656-33-4
Benzene-d5, Nitro-	Nitrobenzene-d5	Nitro(2H5)benzene	4165-60-0
Phen-2,3,4,5-d4-ol, 6-nitro-		2-Nitrophenol-d4	93951-78-1
Phen-2,3,5-d3-ol, 4,6-dichloro-		2,4-Dichlorophenol-d3	93951-74-7
1,2-Benzenedicarboxylic acid, di(methyl-d3)ester		Dimethylphthalate-d6	85448-30-2
Acenaphthylene-d8	Acenaphthylene-d8	Acenaphthylene-d8	93951-97-4
Phen-2,3,5,6-d4-ol, 4-nitro-		4-Nitrophenol-d4	93951-79-2
9H-Fluorene-1,2,3,4,5,6,7,8,9,9-d10	Fluorene-d10	Fluorene-d10	81103-79-9
Phen-3,5-d2-ol, 2-methyl-4,6-dinitro-		4,6-Dinitro-methylphenol-d2	93951-76-9
Anthracene-d10	Anthracene-d10	Anthracene, perdeutero-	1719-06-8
Pyrene-d10	Pyrene-d10	Pyrene-d10	1718-52-1
Benzo[a]pyrene-d12	Benzo[a]pyrene-d12	Benzo[a]pyrene-d12	63466-71-7
Fluoranthene-1,2,3,4,5,6,7,8,9,10-d10		Fluoranthene-d10 (SIM DMC)	93951-69-0
Naphthalene-1,2,3,4,5,6,8-d7, 7(methyl-d3)		2-Methylphthalene-d10 (SIM DMC)	7297-45-2

TABLE 2. DECAFLUOROTRIPHENYLPHOSPHINE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	10.0 - 80.0% of mass 198
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	10.0 - 80.0% of mass 198
197	Less than 2.0% of mass 198
198	Base peak 100% relative abundance (see Note)
199	5.0 - 9.0% of mass 198
275	10.0 - 60.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present but less than mass 443
442	Greater than 50.0% of mass 198
443	15.0 - 24.0% of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may exceed that of m/z 198.

TABLE 3. SEMIVOLATILE DEUTERATED MONITORING COMPOUNDS AND THE ASSOCIATED TARGET ANALYTES

1,4-Dioxane-d_8 (DMC-1)	**Phenol-d_5 (DMC-2)**	**Bis(2-Chloroethyl) ether-d_8 (DMC-3)**
1,4-Dioxane	Benzaldehyde	Bis(2-chloroethyl)ether
	Phenol	2,2'-Oxybis(1-chloropropane)
		Bis(2-chloroethoxy)methane
2-Chlorophenol-d_4 (DMC-4)	**4-Methylphenol-d_8 (DMC-5)**	**4-Chloroaniline-d_4 (DMC-6)**
2-Chlorophenol	2-Methylphenol	4-Chloroaniline
	3-Methylphenol	
	4-Methylphenol	
	2,4-Dimethylphenol	
Nitrobenzene-d_5 (DMC-7)	**2-Nitrophenol-d_4 (DMC-8)**	**2,4-Dichlorophenol-d_3 (DMC-9)**
Acetophenone	Isophorone	2,4-Dichlorophenol
N-Nitroso-di-n-propylamine	2-Nitrophenol	Hexachlorobutadiene
Hexachloroethane		4-Chloro-3-methylphenol
Hexachlorocyclopentadiene		2,4,6-Trichlorophenol
Nitrobenzene		2,4,5-Trichlorophenol
2,6-Dinitrotoluene		1,2,4,5-Tetrachlorobenzene
2,4-Dinitrotoluene		*Pentachlorophenol
N-Nitrosodiphenylamine		2,3,4,6-Tetrachlorophenol
3,3'-Dichlorobenzidine		
Dimethylphthalate-d_6 (DMC-10)	**Acenaphthylene-d_8 (DMC-11)**	**4-Nitrophenol-d_4 (DMC-12)**
Caprolactam	*Naphthalene	2-Nitroaniline
1,1'-Biphenyl	*2-Methylnaphthalene	3-Nitroaniline
Dimethylphthalate	2-Chloronaphthalene	2,4-Dinitrophenol
Diethylphthalate	*Acenaphthylene	4-Nitrophenol
Di-n-butylphthalate	*Acenaphthene	4-Nitroaniline
Butylbenzylphthalate		
Bis(2-ethylhexyl)phthalate		
Di-n-octylphthalate		

TABLE 3. SEMIVOLATILE DEUTERATED MONITORING COMPOUNDS AND THE ASSOCIATED TARGET ANALYTES (CON'T)

Fluorene-d_{10} (DMC-13)	**4,6-Dinitro-2-methylphenol-d_2 (DMC-14)**	**Anthracene-d_{10} (DMC-15)**
Dibenzofuran	4,6-Dinitro-2-methylphenol	Hexachlorobenzene
*Fluorene		Atrazine
4-Chlorophenyl-phenylether		*Phenanthrene
4-Bromophenyl-phenylether		*Anthracene
Carbazole		
Pyrene-d_{10} (DMC-16)	**Benzo(a)pyrene-d_{12} (DMC-17)**	
*Fluoranthene	*Benzo(b)fluoranthene	
*Pyrene	*Benzo(k)fluoranthene	
*Benzo(a)anthracene	*Benzo(a)pyrene	
*Chrysene	*Indeno(1,2,3-cd)pyrene	
	*Dibenzo(a,h)anthracene	
	*Benzo(g,h,i)perylene	

*Included in optional TAL of PAHs and PCP only.

TABLE 4. SEMIVOLATILE DEUTERATED MONITORING COMPOUNDS AND THE ASSOCIATED TARGET ANALYTES FOR OPTIONAL ANALYSIS BY SELECTED ION MONITORING

Fluoranthene-d_{10}	**2-Methylnaphthalene-d_{10}**
Fluoranthene	Naphthalene
Pyrene	2-Methylnaphthalene
Benzo(a)anthracene	Acenaphthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

TABLE 5. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
1,4-Dioxane	0.010	0.010	40.0	±40.0	±50.0
Benzaldehyde	0.100	0.100	40.0	±40.0	±50.0
Phenol	0.080	0.080	20.0	±20.0	±25.0
Bis(2-chloroethyl)ether	0.100	0.100	20.0	±20.0	±25.0
2-Chlorophenol	0.200	0.200	20.0	±20.0	±25.0
2-Methylphenol	0.010	0.010	20.0	±20.0	±25.0
3-Methylphenol	0.010	0.010	20.0	±20.0	±25.0
2,2'-Oxybis-(1-chloropropane)	0.010	0.010	20.0	±25.0	±50.0
Acetophenone	0.060	0.060	20.0	±20.0	±25.0
4-Methylphenol	0.010	0.010	20.0	±20.0	±25.0
N-Nitroso-di-n-propylamine	0.080	0.080	20.0	±25.0	±25.0
Hexachloroethane	0.100	0.100	20.0	±20.0	±25.0
Nitrobenzene	0.090	0.090	20.0	±20.0	±25.0
Isophorone	0.100	0.100	20.0	±20.0	±25.0
2-Nitrophenol	0.060	0.060	20.0	±20.0	±25.0
2,4-Dimethylphenol	0.050	0.050	20.0	±25.0	±50.0
Bis(2-chloroethoxy)methane	0.080	0.080	20.0	±20.0	±25.0
2,4-Dichlorophenol	0.060	0.060	20.0	±20.0	±25.0
Naphthalene	0.200	0.200	20.0	±20.0	±25.0
4-Chloroaniline	0.010	0.010	40.0	±40.0	±50.0
Hexachlorobutadiene	0.040	0.040	20.0	±20.0	±25.0
Caprolactam	0.010	0.010	40.0	±30.0	±50.0
4-Chloro-3-methylphenol	0.040	0.040	20.0	±20.0	±25.0
2-Methylnaphthalene	0.100	0.100	20.0	±20.0	±25.0
Hexachlorocyclopentadiene	0.010	0.010	40.0	±40.0	±50.0
2,4,6-Trichlorophenol	0.090	0.090	20.0	±20.0	±25.0
2,4,5-Trichlorophenol	0.100	0.100	20.0	±20.0	±25.0
1,1'-Biphenyl	0.200	0.200	20.0	±20.0	±25.0
2-Chloronaphthalene	0.300	0.300	20.0	±20.0	±25.0
2-Nitroaniline	0.060	0.060	20.0	±25.0	±25.0
Dimethylphthalate	0.300	0.300	20.0	±20.0	±25.0
2,6-Dinitrotoluene	0.080	0.080	20.0	±20.0	±25.0
Acenaphthylene	0.400	0.400	20.0	±20.0	±25.0
3-Nitroaniline	0.010	0.010	20.0	±25.0	±50.0
Acenaphthene	0.200	0.200	20.0	±20.0	±25.0
2,4-Dinitrophenol	0.010	0.010	40.0	±50.0	±50.0
4-Nitrophenol	0.010	0.010	40.0	±40.0	±50.0
Dibenzofuran	0.300	0.300	20.0	±20.0	±25.0
2,4-Dinitrotoluene	0.070	0.070	20.0	±20.0	±25.0
Diethylphthalate	0.300	0.300	20.0	±20.0	±25.0
1,2,4,5-Tetrachlorobenzene	0.100	0.100	20.0	±20.0	±25.0
4-Chlorophenyl-phenylether	0.100	0.100	20.0	±20.0	±25.0

TABLE 5. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR SEMIVOLATILE ORGANIC COMPOUNDS (CON'T)

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
Fluorene	0.200	0.200	20.0	±20.0	±25.0
4-Nitroaniline	0.010	0.010	40.0	±40.0	±50.0
4,6-Dinitro-2-methylphenol	0.010	0.010	40.0	±30.0	±50.0
4-Bromophenyl-phenyl ether	0.070	0.070	20.0	±20.0	±25.0
N-Nitrosodiphenylamine	0.100	0.100	20.0	±20.0	±25.0
Hexachlorobenzene	0.050	0.050	20.0	±20.0	±25.0
Atrazine	0.010	0.010	40.0	±25.0	±50.0
Pentachlorophenol	0.010	0.010	40.0	±40.0	±50.0
Phenanthrene	0.200	0.200	20.0	±20.0	±25.0
Anthracene	0.200	0.200	20.0	±20.0	±25.0
Carbazole	0.050	0.050	20.0	±20.0	±25.0
Di-n-butylphthalate	0.500	0.500	20.0	±20.0	±25.0
Fluoranthene	0.100	0.100	20.0	±20.0	±25.0
Pyrene	0.400	0.400	20.0	±25.0	±50.0
Butylbenzylphthalate	0.100	0.100	20.0	±25.0	±50.0
3,3'-Dichlorobenzidine	0.010	0.010	40.0	±40.0	±50.0
Benzo(a)anthracene	0.300	0.300	20.0	±20.0	±25.0
Chrysene	0.200	0.200	20.0	±20.0	±50.0
Bis(2-ethylhexyl)phthalate	0.200	0.200	20.0	±25.0	±50.0
Di-n-octylphthalate	0.010	0.010	40.0	±40.0	±50.0
Benzo(b)fluoranthene	0.010	0.010	20.0	±25.0	±50.0
Benzo(k)fluoranthene	0.010	0.010	20.0	±25.0	±50.0
Benzo(a)pyrene	0.010	0.010	20.0	±20.0	±50.0
Indeno(1,2,3-cd)pyrene	0.010	0.010	20.0	±25.0	±50.0
Dibenzo(a,h)anthracene	0.010	0.010	20.0	±25.0	±50.0
Benzo(g,h,i)perylene	0.010	0.010	20.0	±30.0	±50.0
2,3,4,6-Tetrachlorophenol	0.040	0.040	20.0	±20.0	±50.0
Selective Ion Monitoring					
Naphthalene	0.600	0.600	20.0	±25.0	±25.0
2-Methylnaphthalene	0.300	0.300	20.0	±20.0	±25.0
Acenaphthylene	0.900	0.900	20.0	±20.0	±25.0
Acenaphthene	0.500	0.500	20.0	±20.0	±25.0
Fluorene	0.700	0.700	20.0	±25.0	±50.0
Phenanthrene	0.300	0.300	20.0	±25.0	±50.0
Anthracene	0.400	0.400	20.0	±25.0	±50.0
Fluoranthene	0.400	0.400	20.0	±25.0	±50.0
Pyrene	0.500	0.500	20.0	±30.0	±50.0
Benzo(a)anthracene	0.400	0.400	20.0	±25.0	±50.0
Chrysene	0.400	0.400	20.0	±25.0	±50.0
Benzo(b)fluoranthene	0.100	0.100	20.0	±30.0	±50.0
Benzo(k)fluoranthene	0.100	0.100	20.0	±30.0	±50.0
Benzo(a)pyrene	0.100	0.100	20.0	±25.0	±50.0
Indeno(1,2,3-cd)pyrene	0.100	0.100	20.0	±40.0	±50.0

TABLE 5. TECHNICAL ACCEPTANCE CRITERIA FOR INITIAL CALIBRATION, INITIAL CALIBRATION VERIFICATION, AND CONTINUING CALIBRATION VERIFICATION FOR SEMIVOLATILE ORGANIC COMPOUNDS (CON'T)

Analyte	ICV/Opening CCV Minimum RRF	Closing CCV Minimum RRF	Maximum %RSD	ICV/Opening CCV Maximum %D[1]	Closing CCV Maximum %D
Dibenzo(a,h)anthracene	0.010	0.010	25.0	±40.0	±50.0
Benzo(g,h,i)perylene	0.020	0.020	25.0	±40.0	±50.0
Pentachlorophenol	0.010	0.010	40.0	±50.0	±50.0
Deuterated Monitoring Compounds					
1,4-Dioxane-d_8	0.010	0.010	20.0	±25.0	±50.0
Phenol-d_5	0.010	0.010	20.0	±25.0	±25.0
Bis-(2-chloroethyl)ether-d_8	0.100	0.100	20.0	±20.0	±25.0
2-Chlorophenol-d_4	0.200	0.200	20.0	±20.0	±25.0
4-Methylphenol-d_8	0.010	0.010	20.0	±20.0	±25.0
4-Chloroaniline-d_4	0.010	0.010	40.0	±40.0	±50.0
Nitrobenzene-d_5	0.050	0.050	20.0	±20.0	±25.0
2-Nitrophenol-d_4	0.050	0.050	20.0	±20.0	±25.0
2,4-Dichlorophenol-d_3	0.060	0.060	20.0	±20.0	±25.0
Dimethylphthalate-d_6	0.300	0.300	20.0	±20.0	±25.0
Acenaphthylene-d_8	0.400	0.400	20.0	±20.0	±25.0
4-Nitrophenol-d_4	0.010	0.010	40.0	±40.0	±50.0
Fluorene-d_{10}	0.100	0.100	20.0	±20.0	±25.0
4,6-Dinitro-2-methylphenol-d_2	0.010	0.010	40.0	±30.0	±50.0
Anthracene-d_{10}	0.300	0.300	20.0	±20.0	±25.0
Pyrene-d_{10}	0.300	0.300	20.0	±25.0	±50.0
Benzo(a)pyrene-d_{12}	0.010	0.010	20.0	±20.0	±50.0
Fluoranthene-d_{10} (SIM)	0.400	0.400	20.0	±25.0	±50.0
2-Methylnaphthalene-d_{10} (SIM)	0.300	0.300	20.0	±20.0	±25.0

[1] If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

TABLE 6. GAS CHROMATOGRAPH ANALYTICAL CONDITIONS

Carrier Gas:	Helium or Hydrogen 99.999% purity
Column Flow:	30 cm/sec or 1-2 mL/min.
Injector Temperature:	250-300°C
Transfer Line Temperature	250-300°C
Source Temperature	According to manufacturer's specifications
Injection Technique:	On-column
Injection Volume:	1 or 2 µl
Initial Column Temperature Hold	40°C for 4 min.
Column Temperature Program	40-270°C at 10°C/min.
Final Column Temperature Hold	270°C; Hold Required: 3 min. after all analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits, have eluted

TABLE 7. MASS SPECTROMETER ANALYTICAL CONDITIONS

Electron Energy	70 Volts (nominal)
Mass Range	35-500 u
Ionization Mode	Electron Ionization (EI)
Scan Time	Not to exceed 1 sec. per scan

NOTE: For SIM analyses, the Contractor is to use professional judgment and the instrument manufacturer's instructions and guidelines in choosing an appropriate single ion scan or dwell time (usually 50-500 msec per ion).

TABLE 8. CHARACTERISTIC IONS FOR SEMIVOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS

Analyte	Primary Quantitation Ion	Secondary Ion(s)
1,4-Dioxane	88	43,58
Benzaldehyde	77	105,106
Phenol	94	65,66
Bis(2-chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
2-Methylphenol	108	107
3-Methylphenol	108	107
2,2'-Oxybis(1-chloropropane)	45	77,79
Acetophenone	105	77,51
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	107	121,122
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
1,1'-Biphenyl	154	153,76
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,182
2,6-Dinitrotoluene	165	89,121
Diethylphthalate	149	177,150
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
4-Chlorophenyl-phenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108
4,6-Dinitro-2-methylphenol	198	182,77
N-Nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenylether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215

TABLE 8. CHARACTERISTIC IONS FOR SEMIVOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS, AND INTERNAL STANDARDS (CON'T)

Analyte	Primary Quantitation Ion	Secondary Ion(s)
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	101,100
Pyrene	202	101,100
Butylbenzylphthalate	149	91,206
3,3'-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	226,229
Di-n-octyl phthalate	149	none
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenzo(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Deuterated Monitoring Compounds		
1,4-Dioxane-d_8	96	64,34
Phenol-d_5	99	71,42
Bis(2-chloroethyl)ether-d_8	67	99,69
2-Chlorophenol-d_4	132	134,68,66
4-Methylphenol-d_8	113	115,54
4-Chloroaniline-d_4	131	133,69
Nitrobenzene-d_5	128	82,54
2-Nitrophenol-d_4	143	69,41,42
2,4-Dichlorophenol-d_3	165	167,101
Dimethylphthalate-d_6	166	78
Acenaphthylene-d_8	160	80,158
4-Nitrophenol-d_4	143	113,41,42
Fluorene-d_{10}	176	174,87,86
4,6-Dinitro-2-methylphenol-d_2	200	170,52
Anthracene-d_{10}	188	94,80
Pyrene-d_{10}	212	106,104
Benzo(a)pyrene-d_{12}	264	132,118
Fluoranthene-d_{10}(SIM)	212	106,104
2-Methylnaphthalene-d_{10} (SIM)	152	151
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162,160
Phenanthrene-d_{10}	188	94,80
Chrysene-d_{12}	240	120,236
Perylene-d_{12}	264	260,265

TABLE 9. SEMIVOLATILE INTERNAL STANDARDS WITH ASSOCIATED TARGET AND DEUTERATED MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
1,4-Dioxane Benzaldehyde Phenol Bis(2-chloroethyl) ether 2-Chlorophenol 2-Methylphenol 3-Methylphenol 2,2'-Oxybis(1-chloro-propane) Acetophenone 4-Methylphenol N-Nitroso-di-n-propylamine Hexachloroethane 1,4-Dioxane-d_8 (DMC) Phenol-d_5 (DMC) Bis(2-chloroethyl)ether-d_8 (DMC) 2-Chlorophenol-d_4 (DMC) 4-Methylphenol-d_8 (DMC)	Nitrobenzene Isophorone 2-Nitrophenol 2,4-Dimethylphenol Bis(2-chloroethoxy)methane 2,4-Dichlorophenol Hexachlorobutadiene Caprolactam 4-Chloro-3-methylphenol *2-Methylnaphthalene *Naphthalene 4-Chloroaniline Nitrobenzene-d_5 (DMC) 2-Nitrophenol-d_4 (DMC) 2,4-Dichlorophenol-d_3 (DMC) 4-Chloroaniline-d_4 (DMC) 2-Methylnaphthalene-d_{10} (SIM-DMC)	Hexachlorocyclopentadiene 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 2,3,4,6-Tetrachlorophenol 1,1'-Biphenyl 2-Chloronaphthalene 2-Nitroaniline Dimethylphthalate *Acenaphthylene 3-Nitroaniline *Acenaphthene 2,4-Dinitrophenol 4-Nitrophenol Dibenzofuran 2,4-Dinitrotoluene 2,6-Dinitrotoluene 1,2,4,5-Tetrachlorobenzene Diethylphthalate 4-Chlorophenyl-phenylether *Fluorene 4-Nitroaniline Acenaphthylene-d_8 (DMC) 4-Nitrophenol-d_4 (DMC) Dimethylphthalate-d_6 (DMC) Fluorene-d_{10} (DMC)
Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
4,6-Dinitro-2-methylphenol N-Nitrosodiphenylamine 4-Bromophenyl-phenylether Hexachlorobenzene Atrazine *Pentachlorophenol *Phenanthrene *Anthracene Carbazole Di-n-butylphthalate *Fluoranthene 4,6-Dinitro-2-methylphenol-d_2 (DMC) Anthracene-d_{10} (DMC) Fluoranthene-d_{10} (SIM-DMC)	*Pyrene Butylbenzylphthalate 3,3'-Dichlorobenzidine *Benzo(a)anthracene Bis(2-ethylhexyl)phthalate *Chrysene Pyrene-d_{10} (DMC)	Di-n-octylphthalate *Benzo(b)fluoranthene *Benzo(k)fluoranthene *Benzo(a)pyrene *Indeno(1,2,3-cd)pyrene *Dibenzo(a,h)anthracene *Benzo(g,h,i)perylene Benzo(a)pyrene-d_{12} (DMC)

* Included in optional TAL of PAHs and PCP only.

TABLE 10. INTERNAL STANDARDS WITH ASSOCIATED TARGET AND DEUTERATED MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION OF POLYNUCLEAR AROMATIC HYDROCARBON AND PENTACHLOROPHENOL

Naphthalene-d_8	**Acenaphthene-d_{10}**	**Phenanthrene-d_{10}**
2-Methylnaphthalene Naphthalene 2,4-Dichlorophenol-d_3 (DMC) *2-Methylnaphthalene-d_{10} (DMC)	Acenaphthylene Acenaphthene Fluorene Acenaphthylene-d_8 (DMC) Fluorene-d_{10} (DMC)	Phenanthrene Anthracene Fluoranthene Pentachlorophenol Anthracene-d_{10} (DMC) *Fluoranthene-d_{10} (DMC)
Chrysene-d_{12}	**Perylene-d_{12}**	
Pyrene Benzo(a)anthracene Chrysene Pyrene-d_{10} (DMC)	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene Benzo(a)pyrene-d_{12} (DMC)	

* DMC assigned only for PAH and PCP by SIM analysis.

TABLE 11. DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

Compound	Percent Recovery For Water Samples	Percent Recovery For Soil Samples
1,4-Dioxane-d_8	40-110	40-110
Phenol-d_5	10-130	10-130
Bis(2-chloroethyl)ether-d_8	25-120	10-150
2-Chlorophenol-d_4	20-130	15-120
4-Methylphenol-d_8	25-125	10-140
4-Chloroaniline-d_4	1-146*	1-145*
Nitrobenzene-d_5	20-125	10-135
2-Nitrophenol-d_4	20-130	10-120
2,4-Dichlorophenol-d_3	20-120	10-140
Dimethylphthalate-d_6	25-130	10-145
Acenaphthylene-d_8	10-130	15-120
4-Nitrophenol-d_4	10-150	10-150
Fluorene-d_{10}	25-125	20-140
4,6-Dinitro-2-methylphenol-d_2	10-130	10-130
Anthracene-d_{10}	25-130	10-150
Pyrene-d_{10}	15-130	10-130
Benzo(a)pyrene-d_{12}	20-130	10-140
Fluoranthene-d_{10} (SIM)	30-130	30-130
2-Methylnaphthalene-d_{10} (SIM)	30-130	20-140

* Limits are advisory.

TABLE 12. MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Percent Recovery Water	RPD Water	Percent Recovery Soil	RPD Soil
Phenol	12-110	0-42	26-90	0-35
2-Chlorophenol	27-123	0-40	25-102	0-50
N-Nitroso-di-n-propylamine	41-116	0-38	41-126	0-38
4-Chloro-3-methylphenol	23-97	0-42	26-103	0-33
Acenaphthene	46-118	0-31	31-137	0-19
4-Nitrophenol	10-80	0-50	11-114	0-50
2,4-Dinitrotoluene	24-96	0-38	28-89	0-47
Pentachlorophenol	9-103	0-50	17-109	0-47
Pyrene	26-127	0-31	35-142	0-36

EXHIBIT D

PESTICIDES ANALYSIS

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EXHIBIT D - Pesticides Analysis

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EXHIBIT D - Pesticides Analysis

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1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water, leachate derived from the Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP), and soil/sediment samples from hazardous waste sites to determine the presence and concentration of the chlorinated pesticides contained in the Target Analyte List (TAL) for pesticides in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. The method, based on U.S. Environmental Protection Agency (EPA) SW-846 Method 8081C, can be used for determining analyte concentrations in the range from the Contract Required Quantitation Limits (CRQLs) to one million times the CRQL in these matrices, when appropriate dilutions are made. The method includes sample extraction, extract cleanup techniques, and Gas Chromatograph/Electron Capture Detector (GC/ECD) analytical methods for chlorinated pesticides.

1.2 Co-elution problems have been associated with the following pairs of analytes using this method include:

- On a DB-608 or equivalent column, DDE and Dieldrin; Methoxychlor and Endrin ketone; and Endosulfan I and trans-Chlordane; and
- On a DB-1701 or equivalent column, Endosulfan I and trans-Chlordane, and Methoxychlor and Endosulfan sulfate.

1.3 There are two isomers of heptachlor epoxide, the endo epoxy isomer (Isomer A) and the exo epoxy isomer (Isomer B). The two isomers are separable using current GC capillary columns. Only the exo epoxy isomer (Isomer B) is of environmental significance. This is the isomer that must be used as an analytical standard, identified and quantitated in sample analysis, and reported on appropriate forms as heptachlor epoxide.

2.0 SUMMARY OF METHOD

2.1 Water/TCLP or SPLP Leachate

Continuous liquid-liquid extraction or separatory funnel extraction procedures are employed for aqueous samples. A 1.0 Liter (L) aliquot of sample is spiked with the surrogate solution and extracted with methylene chloride using a separatory funnel or a continuous extractor. The methylene chloride extract is dried with anhydrous sodium sulfate (or Hydromatrix™), concentrated, and subjected to Gel Permeation Chromatography (GPC) cleanup. GPC is required when higher molecular weight compounds are present that interfere with the analyses of target analytes; GPC is optional for all other circumstances. The extract is then solvent exchanged into hexane, cleaned up by Florisil cartridges or other methods as applicable, and analyzed using a dual column wide-bore capillary GC/ECD.

2.2 Soil/Sediment

A 30 gram (g) aliquot of sample is spiked with the surrogates, mixed with anhydrous sodium sulfate (or Hydromatrix™), and extracted with a 1:1 (v/v) acetone/methylene chloride solvent mixture by ultrasonic extraction, Soxhlet extraction, or pressurized fluid extraction. The extract is filtered (for ultrasonic extraction), concentrated, and solvent-exchanged into methylene chloride. The methylene chloride extract is then subjected to GPC (mandatory), solvent-exchanged into hexane, cleaned up by a Florisil cartridge or other methods as

applicable, and analyzed using a dual column wide-bore capillary GC/ECD.

2.3 Wipes

Not applicable to this method.

2.4 Waste

Not applicable to this method.

3.0 DEFINITIONS

See Exhibit G – Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

Method interferences may be caused by compounds in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts and/or to elevated baselines in Gas Chromatograms. These materials must be routinely demonstrated to be free from interferences under the sample preparation and analysis conditions by analyzing instrument and method blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Because common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.2 Matrix Interferences

Matrix interferences may be caused by compounds that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. The cleanup procedures in this method must be used to remove such interferences in order to achieve the CRQLs.

5.0 SAFETY

See Section 12.0 of Exhibit D – Introduction to Organic Analytical Methods.

5.1 Reagents

Concentrated sulfuric acid presents some hazards and is moderately toxic and extremely irritating to skin and mucous membranes. Use this reagent in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with this reagent.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternative equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 General Laboratory Equipment

6.1.1 Balances

6.1.1.1 Top loading, capable of weighing accurately to ±0.01 g.

6.1.1.2 Analytical, capable of weighing accurately to ±0.0001 g.

6.1.1.3 A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each type of balance and be accurate to ±0.01 g and ±0.0001 g, respectively. The masses that are used to check the balances daily must be checked on a monthly basis using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '0' or Class '1') as defined by ASTM E617-97(2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified at least every five years, or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates these criteria have been met.

6.1.2 Beakers - 100 milliliter (mL), 125 mL, 250 mL, and 400 mL.

6.1.3 Centrifuge, Table top (optional).

6.1.3.1 Centrifuge Tube - 12-15 mL with 19 millimeter (mm) ground-glass joint (optional).

6.1.4 Graduated Cylinder Class A – 1.0 L and 100 mL capacity.

6.1.5 Desiccator.

6.1.6 Erlenmeyer Flasks - 250 mL.

6.1.7 Volumetric Flask, Class A – 5.0, 10, 20, 50, 100, 250, and 500 mL.

6.1.8 Magnetic Stirring Bar - Polytetrafluoroethylene (PTFE) coated, at least 4 centimeters (cm) long.

6.1.9 Ovens – drying, capable of maintaining 105°C (+/- 5°C).

6.1.10 pH Meter - With a combination glass electrode. Calibrate according to manufacturer's instructions. The pH meter shall be calibrated prior to each use, using reference standards bracketing the range expected in samples. The pH reference standards shall be replaced when their expiration dates have passed.

6.1.11 pH Paper – Wide range.

6.1.12 Pipettes (Calibrated) - Glass volumetric, 1.0 mL or 2.0 mL. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.1.13 Spatula - Stainless steel or PTFE.

6.1.14 Syringes – 10 microliters (µL), 25 µL, 100 µL, and 1000 µL.

6.1.15 Vials and Caps - 10 mL (optional), with screw-cap and PTFE or aluminum foil liner; autosampler vial with 2 mL capacity for GC autosampler.

6.1.16 Weigh Dish – Porcelain crucibles or disposable aluminum weighing pans.

6.2 Glassware/Extraction/Cleanup Equipment

6.2.1 Automated Soxhlet Extraction System - With temperature-controlled oil bath. Silicone oil must not be used because it destroys the rubber parts. The apparatus must be used in a hood.

6.2.1.1 Cellulose or Glass Extraction Thimble, 26 mm x 60 mm.

6.2.1.2 Glass Extraction Cups.

6.2.1.3 Thimble Adapters.

6.2.1.4 Viton Seals.

6.2.2 Soxhlet Extraction, Manual

6.2.2.1 Allihn Condenser.

6.2.2.2 Soxhlet Extractor body, 40 mm ID.

6.2.2.3 Round bottom flask, 500 mL.

6.2.3 Borosilicate Glass Wool - Rinsed with methylene chloride.

6.2.4 Continuous Liquid-Liquid Extractors - Equipped with PTFE or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf extractor) or hydrophobic membrane-based extractor.

6.2.5 Drying Column – 400 mm x 19 mm ID chromatographic column with coarse frit (substitution of a small pad of disposable borosilicate glass wool for the frit will help prevent cross-contamination of sample extracts).

6.2.6 Florisil Cleanup Equipment

6.2.6.1 Florisil - 500 milligram (mg) or 1 g cartridges with stainless steel or PTFE frits.

6.2.6.2 Vacuum System for Eluting Multiple Cleanup Cartridges.

6.2.6.3 Vacuum Trap - Made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

6.2.6.4 Vacuum Pressure Gauge.

6.2.7 Gel Permeation Chromatography Equipment

6.2.7.1 GPC System - Systems that perform satisfactorily have been assembled from the following components: a High Performance Liquid Chromatography (HPLC) pump; an autosampler or a valving system with sample loops; and a fraction collector. All systems, whether automated or manual, must meet the calibration requirements in Section 10.3.1.3.

NOTE: GPC cleanup is required for all soil/sediment extracts, and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target analytes.

6.2.7.2 Chromatographic Column - 700 mm x 25 mm ID glass column. Flow is upward. To simplify switching from the ultraviolet (UV) detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

6.2.7.3 Guard Column (optional) - 5 cm, with appropriate fittings to connect to the inlet side of the analytical column.

6.2.7.4 Bio Beads (SX-3) - 200 to 400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, or equivalent). An additional 5 g of Bio Beads is required if the optional guard column is employed. The quality of Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they can also pass through the column screens and damage the valve.

6.2.7.5 UV Detector - Fixed wavelength [254 nanometers (nm)] with a semi-prep flow-through cell.

6.2.7.6 Strip Chart Recorder - Recording integrator or laboratory data system.

6.2.7.7 Syringe Filter Assembly, disposable - 5 micron filter discs.

NOTE: Consult your instrument operation manual to determine the proper filter disc to use in your system. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

6.2.7.8 Viscometer

6.2.8 Kuderna-Danish (K-D) Apparatus

6.2.8.1 Concentrator Tubes - 10 mL and 15 mL, graduated.

6.2.8.2 Evaporative Flasks - 500 mL.

6.2.8.3 Silicon Carbide Boiling Chips – Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride. PTFE boiling chips solvent rinsed prior to use are acceptable.

6.2.8.4 Snyder Column - Three-ball macro.

6.2.8.5 Snyder Column - Two-ball micro.

6.2.9 Nitrogen Evaporation Device - Equipped with a water bath that can be maintained at 35-40°C. To prevent the release of solvent fumes into the laboratory, the nitrogen evaporator device must be used in a hood.

6.2.10 Pressurized Fluid Extraction Device

6.2.10.1 Dionex Accelerated Solvent Extractor (ASE-300) or equivalent with appropriately-sized extraction cells. Currently, 100 mL cells are available that will accommodate greater than 30 g samples. Cells should be made of stainless steel or other material capable of withstanding the pressure requirements [2000+ pounds per square inch (psi)] necessary for this procedure.

6.2.10.2 Other system designs may be employed, provided that adequate performance can be demonstrated for the analytes and matrices of interest.

6.2.11 Separatory Funnels - 2 L with PTFE stopcock.

6.2.12 Sonabox Acoustic Enclosure (or equivalent) - For use with disruptor to decrease noise level.

6.2.13 Ultrasonic Cell Disruptor - QSonica LLC, (53 Church Hill Road, Newtown, CT 06470) model S-4000 or equivalent ultrasonic liquid disruptor – equipped with a 3/4-inch horn and a 1/2-inch horn with a minimum output capacity of 300 watts.

NOTE 1: To ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. A rough tip surface is an indication of erosion.

NOTE 2: Follow manufacturer's instructions for set-up.

6.2.14 Vacuum or Pressure Filtration Apparatus

6.2.14.1 Buchner Funnel.

6.2.14.2 Filter Paper – Whatman No. 42, or equivalent.

6.2.15 Water Bath - Heated, with concentric ring cover, capable of temperature control. The bath should be used in the hood.

6.3 Analytical Instrumentation

6.3.1 Gas Chromatograph

The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout the temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases. The instrument must be vented to outside the facility or to a trapping system that prevents the release of contaminants into the instrument room. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants or flow controllers with rubber components are not to be used.

6.3.1.1 GCs may have difficulty in meeting certain method Quality Control (QC) requirements of Endrin and DDT breakdown in the injector. This problem can be minimized by operating the injector at 200-205°C, using a borosilicate glass (not quartz) methyl silicone deactivated injector liner, and deactivating the metal parts in the injector with dichlorodimethylsilane. In some cases, using a 0.25-inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

6.3.2 Gas Chromatography Columns

Recommended Columns: Wide-bore (0.53 mm ID) fused silica GC columns may be used provided that the resolution requirements are met (Section 9.3.5.2); if two wide-bore (0.53 mm ID) fused silica GC columns are used, then a separate detector is required for each column. The specified analytical columns are a 30 m x 0.53 mm ID, 1.0 µm film thickness DB-1701 (J&W Scientific); SPB 1701 (Supelco); AT 1701 (Alltech); Rtx®-1701, Rtx® CLP I (Restek); CP-Sil 19CB (Chrompack); 007-1701 (Quadrex); BP-10 (SGE); or equivalent, and a 30 m x 0.53 mm ID, 0.5 to 1.0 µm film thickness DB-608 (J&W Scientific); HP-608 (Agilent); SPB-608 (Supelco); 007-608 (Quadrex); BP-608 (SGE); Rtx® CLP II; CP-Sil 8CB (Chrompack); or equivalent. A description of the GC columns used for analysis shall be provided in the SDG Narrative. Packed columns may not be used.

6.3.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants that interfere with the identification and quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated using the column meet the initial calibration and continuing calibration verification (CCV) technical acceptance criteria listed in the analytical method in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits;
- The column can accept at least 16 times the low-point initial calibration concentration level in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides, without becoming overloaded; and
- The column pair chosen must have dissimilar phases/chemical properties in order to separate the analytes of interest in different Retention Time (RT) order.

6.3.2.1.1 The column provides equal or better resolution of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits, than the columns listed in Section 6.3.2. Although the instructions included in the analytical method are for wide-bore capillary columns, narrower bore capillary columns may be evaluated for use. Follow manufacturer's instructions for use of its product. Document in the SDG Narrative if other columns are used by specifying the column used.

6.3.2.1.2 The Contractor must maintain documentation verifying that the alternate column met the criteria in Section 6.3.2.1. The minimum documentation is as follows:

6.3.2.1.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.3.2.1.2.2 Chromatograms and data system reports generated on the GC/ECD and used for EPA Contract Laboratory Program (CLP) analyses, including those from:

- Instrument blanks demonstrating there are no contaminants that interfere with the pesticides analysis when using the alternate column; and
- The analysis of initial calibration and CCV standards using the alternate column.

6.3.2.1.3 Based on the Contractor-generated data described above, the Contractor shall complete a written review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Section 6.3.2;
- The low-point initial calibration standard analyses have adequate sensitivity to meet the pesticide CRQLs;
- The high-point initial calibration standard analyses were not overloaded; and

- The alternate column does not introduce contaminants that interfere with the identification and/or quantitation of analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

6.3.2.1.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request by the EPA Regional CLP Contracting Officer's Representative (COR).

6.3.2.1.5 Columns may be mounted in a press-fit Y-shaped glass 3-way union splitter or a Y-shaped fused-silica connector from a variety of commercial sources. The two columns may be mounted in an 8-inch deactivated glass injection tee. The Contractor should follow the manufacturer's recommendations for mounting 0.53 mm capillary columns in injector ports. Optionally, the dual column GC with separate autosamplers can be used for sample extract injection.

6.3.2.1.6 The carrier gas for routine applications is helium. The Contractor may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the SDG Narrative in submissions to the EPA. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants or flow controllers with rubber components are not to be used.

6.3.2.2 Gel Permeation Chromatography Column Preparation

Prepare the GPC column using Bio Beads. Alternate column packings may be use if: 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration verification, and 2) the column packings do not introduce contaminants/artifacts into the sample that interfere with the analysis of the pesticide analytes. Follow the manufacturer's instructions for preparation of the GPC column.

6.3.3 Electron Capture Detector

6.3.3.1 The linearity of the response of the ECD may be greatly dependent on the flow rate of the make-up gas. The make-up gas must be P-5, P-10 (argon/methane), or nitrogen according to the instrument specification. Care must be taken to maintain stable and an appropriate flow of make-up gas to the detector. The GC/ECD system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants that may interfere with the analysis. The instrument must be vented to outside the facility or to a trapping system that prevents the release of contaminants into the instrument room.

6.3.3.2 At least annually, each ECD should be checked for radiation leakage from their Ni-63 source. Wipe tests should be conducted by wiping the inlet, outlet, and body of the ECD cell with swabs and sending the swabs for radiation tests.

6.4 Data Systems/Data Storage

A data system must be interfaced to the GC/ECD that allows the continuous acquisition and storage of data from each column throughout the duration of the chromatographic program and must permit, at a minimum, the output of time vs. intensity (peak height or peak area) data. The data system must be able to rescale chromatographic data in order to report chromatograms meeting the requirements listed within this method.

7.0 REAGENTS AND STANDARDS

The Contractor must provide all standards to be used with the contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Organic Analytical Methods, Section 11. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1 Reagents

7.1.1 Reagent Water - Reagent water is defined as water in which an interferent is not observed at or above the CRQL for each analyte of interest.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g [1 pound (lb)] of activated carbon.

7.1.1.2 Reagent water may also be generated using a water purification system.

7.1.2 10% acetone in hexane (v/v) - Prepare by adding 10.0 mL of acetone to 90.0 mL of hexane.

7.1.3 Acetone/methylene chloride (1:1 v/v).

7.1.4 Copper powder (optional) - Fine, granular. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

7.1.5 Hydromatrix™ - Diatomaceous earth-based material rinsed with methylene chloride and dried at 400°C for 4 hours in a shallow tray, cooled in a desiccator, and stored in a glass bottle.

7.1.6 Nitric Acid - Dilute, for sulfur removal with copper.

7.1.7 Sodium Hydroxide Solution (10 N) – Carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 mL.

7.1.8 Sodium sulfate - Granular anhydrous reagent grade, heated at 400°C for 4 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by a GC/ECD to demonstrate that it is free of interference before use or must be purchased with certification that it is free of interference.

CAUTION: AN OPEN CONTAINER OF SODIUM SULFATE MAY BECOME CONTAMINATED DURING STORAGE IN THE LABORATORY.

7.1.9 Sodium sulfite.

7.1.10 Solvents: Methylene chloride, hexane, acetone, toluene, iso-octane, petroleum ether, and methanol (optional) - pesticide quality or equivalent. It is recommended that each lot of solvent be analyzed to demonstrate that it is free of interference before use or must be purchased with certification that it is free of interference. Methylene chloride must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid. Acidic methylene chloride must be passed through basic alumina and then demonstrated to be free of hydrochloric acid.

7.1.11 Sulfuric acid, concentrated, 95-98% (sp. gr. 1.84).

7.1.12 Tetrabutylammonium sulfite.

7.1.13 Glycerol.

7.2 Standards

7.2.1 Stock Standard Solutions

7.2.1.1 Stock standard solutions are defined as standards that are to be used to produce working standards. They may be in the form of single compounds or mixtures. They may be prepared in methylene chloride from pure standard materials, or purchased as pre-made solutions. Prepare fresh stock standards every 6 months, or sooner if the standard has degraded or evaporated.

7.2.2 Working Standards

7.2.2.1 Individual Standard Mixtures

7.2.2.1.1 The Calibration Standard Mixture solutions must be prepared in either hexane or iso-octane. The analysis of the Resolution Check Mixture will determine whether one or two sets of Individual Standard Mixture solutions will be needed.

7.2.2.1.2 The suggested compositions of Individual Standard Mixture A and Mixture B are listed in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides, with the concentrations of each target analyte and surrogate given for the low-point standard mixtures (CS1 Standard A and CS1 Standard B) in Table 4 – Low Concentration Calibration Standard (CS1) for Individual Standard Mixtures A and B. The CS1 Standard C for Individual Standard Mixture C will contain all target analytes and surrogates for both Mixture A and Mixture B at the same concentrations as the CS1 Standard for Mixture A and Mixture B.

7.2.2.1.3 Prepare calibration standards at a minimum of five concentration levels. The concentrations of the pesticides in the low-point standard mixtures (CS1) correspond to the low-point concentration (refer to Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides) or lower for each analyte. The concentration for each analyte in the high-point standard must be at least 16 times the concentration of the low-point standard, but a higher concentration may be chosen by the Contractor provided that the higher concentration standards meet the technical acceptance criteria in Sections 9.3.5 and 9.4.5.

7.2.2.1.4 The concentration of each target analyte for each calibration standard are listed in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides. These levels are based upon 10 mL final volume extracts for samples not undergoing GPC cleanup, and 5.0 mL final volume extracts for those samples undergoing GPC cleanup.

7.2.2.1.5 Other concentration levels may be used for more sensitive instrumentation and final extract volumes. For example, in the case of alpha-BHC, a laboratory may use a final extract volume of 10 mL for samples undergoing GPC cleanup, and a low calibration standard of 2.5 nanograms (ng)/mL. The alternate calibration standards and final volumes may be used as long as the following requirements are met:

- The Contractor can demonstrate by Method Detection Limit (MDL) studies that the MDL study calculated MDL for each target analyte is below the required CRQL for that analyte when using the laboratory's specific final volume and calibration level scheme.
- All five calibration levels are in the same ratio as that shown in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides (e.g., if a laboratory were using a 2.5 ng/mL low standard, then the other calibration levels must be 5, 10, 20, and 40 ng/mL).

7.2.2.2 Toxaphene Standards

Prepare Toxaphene standard solutions at a minimum of five concentration levels. The Toxaphene standards must be prepared in hexane or iso-octane and contain the surrogates at the appropriate concentrations.

7.2.2.2.1 For CS1, the concentrations of tetrachloro-m-xylene and decachlorobiphenyl should be 5 and 10 ng/mL, respectively.

7.2.2.2.2 The concentration of Toxaphene in the low-point standard (CS1) should be 500 ng/mL or lower. The concentration in the high-point standard (CS5) must be at least 16 times the low-point standard for Toxaphene, but a higher concentration may be chosen by the Contractor. For most operations, the calibration standards are to be prepared at 500, 1000, 2000, 4000, and 8000 ng/mL (for calibration standards and final volumes, see Section 7.2.2.1.2).

7.2.2.2.3 The low-point Toxaphene standard (CS1) in Section 7.2.2.2.2 shall be used as the single-point initial calibration standard. When Toxaphene is detected in a sample, a five-point toxaphene initial calibration must be initiated on the GC/ECD and the sample containing the Toxaphene must be reanalyzed.

7.2.2.3 Continuing Calibration Standard

7.2.2.3.1 The CCV Standards INDA and INDB or INDC should contain the target analytes and surrogates at or near the mid-point CS3 concentration of the Initial Calibration Standard (ICAL) (Table 2 - Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Pesticides).

7.2.2.4 Instrument Performance Check Standards

7.2.2.4.1 Resolution Check Mixture

Prepare the Resolution Check Mixture containing the pesticides and surrogates listed in Table 3 – Instrument Performance Check Standards, in hexane or iso-octane at the concentrations specified. The mixture must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.4.2 Performance Evaluation Mixture

Prepare the Performance Evaluation Mixture (PEM) solution containing the pesticides and surrogates listed in Table 3 – Instrument Performance Check Standards, in hexane or iso-octane at the concentration specified. The PEM must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.5 Gel Permeation Chromatography Calibration and Calibration Verification Solutions

7.2.2.5.1 GPC Calibration Solution

Prepare a GPC calibration solution in methylene chloride that contains the following analytes at the minimum concentrations listed below. The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

Analyte	Concentration (mg/mL)
Corn oil (CAS# 8001-30-7)	25.0
Bis(2-ethylhexyl)phthalate (CAS# 117-81-7)	0.50
Methoxyclor (CAS# 72-43-5)	0.10
Perylene (CAS# 198-55-0)	0.020
Sulfur (CAS# 7704-34-9)	0.080

NOTE: Sulfur is not very soluble in methylene chloride, but it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds.

7.2.2.5.2 GPC Calibration Verification Solution

Prepare the GPC calibration verification solution containing the pesticides listed in Table 7 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in methylene chloride at the concentrations specified for a 5 mL GPC injection loop. See Section 10.3.1.4.3 for analyte concentrations if a smaller size loop is being used. The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.6 Florisil Cartridge Check Solution

Prepare a solution containing 2,4,5-trichlorophenol at 0.10 µg/mL in acetone. The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.7 Surrogate Standard Spiking Solution

The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added prior to extraction to all standards, samples [including Laboratory Control Samples (LCSs)], Matrix Spike/Matrix Spike Duplicates (MS/MSDs), Performance Evaluation (PE) samples (if required), and required blanks (method/sulfur cleanup/ instrument). Prepare a surrogate spiking solution of 0.20 µg/mL for tetrachloro-m-xylene and 0.40 µg/mL for decachlorobiphenyl in acetone. The solution should be checked frequently for stability. The solution must be replaced every 6 months, or sooner if the solution has degraded or concentrated.

NOTE: Other concentrations for surrogate standard spiking solutions may be used, provided that the appropriate amount of each surrogate is added to all standards, samples (including LCSs), MS/MSDs, PE samples, and blanks.

7.2.2.8 Matrix Spiking Solution

Prepare a matrix spiking solution containing the pesticides in Table 7 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in methanol at the concentrations specified. The solution must be replaced every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.9 Laboratory Control Sample Spiking Solution

Prepare an LCS spiking solution containing the analytes as specified in Table 7 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in methanol. The LCS solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.3 Storage of Standards

7.2.3.1 Store the stock standard solutions at ≤ 6°C, but not frozen, in PTFE-lined, screw-cap, amber bottles/vials.

7.2.3.2 The working standards must be prepared every 6 months, or sooner if the solutions have degraded or concentrated. The working standards must be checked frequently for signs of degradation or evaporation. Store the working standard solutions at ≤ 6°C in PTFE-lined screw-cap, amber bottles/vials.

NOTE: Refrigeration of GPC calibration solutions may cause the corn oil to precipitate. Before use, allow the solution to stand at room temperature until the corn oil dissolves. Replace this calibration solution every 6 months, or more frequently if necessary.

7.2.3.3 Standard solutions purchased from a chemical supply company as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no manufacturer’s expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor that are immediately ampulated in glass vials may be retained for 2 years from the preparation date. The expiration date of the ampulated standards, upon the breaking of the glass seal, is 6 months (or sooner if the standard has degraded or evaporated).

7.2.3.4 Protect all standards from light.

7.2.3.5 Samples, sample extracts, and standards must be stored separately.

7.2.3.6 The Contractor is responsible for maintaining and verifying the integrity of standard solutions prior to use. Storage of standard solutions in the freezer may cause some compounds to precipitate. This means at a minimum, the standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution. Additional steps may be necessary to ensure all components are in solution.

7.2.4 Temperature Records for Storage of Standards

7.2.4.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators/freezers.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Water samples

Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with PTFE-lined screw-caps. If amber containers are not available, the samples should be protected from light.

8.1.2 Soil/Sediment Samples

Soil/sediment samples may be collected in glass containers.

8.2 Procedure for Sample and Sample Extract Storage

8.2.1 Sample Storage

The samples must be protected from light and refrigerated at ≤ 6°C, but not frozen, from the time of receipt until 60 days after delivery of a complete, reconciled data package to the EPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 Sample Extract Storage

Sample extracts must be protected from light and stored at ≤6 °C, but not frozen, until 365 days after delivery of a complete, reconciled data package to the EPA.

8.3 Contract Required Holding Times

8.3.1 Extraction of water samples by separatory funnel procedures must be completed within 5 days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction must be started within 5 days of VTSR. Extraction of the TCLP or SPLP filtrates and leachates shall begin within 7 days of completion of the filtration and leaching procedures. Extraction of soil/sediment samples shall be completed within 10 days of VTSR.

8.3.2 Analysis of sample extracts must be completed within 40 days following the start of extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Initial Instrument Set-up

9.1.1 Gas Chromatograph

9.1.1.1 The GC analytical conditions are provided in Table 6 – Gas Chromatograph Analytical Conditions. Other conditions may be used, provided that all technical acceptance criteria in Sections 9.3.5, 9.4.5, and 11.3 are met.

9.1.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples (including LCSs and MS/MSDs), and required blanks (method/sulfur cleanup/instrument).

9.1.1.3 The same injection volume, 1.0 or 2.0 µL, must be used for all standards, samples (including LCSs and MS/MSDs), and required blanks (method/sulfur cleanup/instrument).

9.1.1.4 The linearity of the ECD may be greatly dependent on the flow rate of the make-up gas. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector.

9.1.1.5 Cold (ambient temperature) on-column injectors that allow injection directly onto a 0.53 mm ID column may be used as long as the initial calibration and calibration verification technical acceptance criteria are met.

9.2 Instrument Performance Check

The instrument performance checks include the Resolution Check Standard (RESC), and the PEM, which are incorporated into the calibration procedures below. Target analyte resolution and stability are verified by the analysis of these instrument performance checks.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to analysis of samples (including LCSs and MS/MSDs) and required blanks (method/sulfur cleanup/instrument), each GC/ECD system must be calibrated at a minimum of five concentrations for single component analytes and surrogates, in order to determine instrument sensitivity and the linearity of GC response. For Toxaphene detected using a single-point calibration, a reanalysis of the sample is required after a five-point calibration.

9.3.2 Frequency of Initial Calibration

Each GC/ECD system must be calibrated prior to analyzing samples, after major instrument maintenance or modification is performed (e.g., column replacement or repair, cleaning or replacement of the ECD, etc.), or if the CCV technical acceptance criteria have not been met.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set up the GC/ECD system as described in Section 9.1. Optimize the instrumental conditions for resolution of the target analytes and sensitivity.

NOTE: Once the GC conditions have been established, the same operating conditions must be used for both calibrations and sample analyses.

9.3.3.2 Prepare the initial calibration standards using the procedures, analytes, and concentrations specified in Section 7.2.2.

9.3.3.3 All standards and instrument blanks must be allowed to warm to ambient temperature before analysis.

9.3.3.4 The initial calibration sequence shall begin with a Resolution Check Mixture, followed by a PEM. The sequence shall end with analysis of an Instrument Blank, followed immediately with a PEM. The appropriate calibration sequence is determined by the results of the Resolution Check Mixture (Section 9.3.4 and 9.3.5.2). All steps pertaining to the initial calibration sequence shall be performed uninterrupted with no more than the length of one chromatographic run separating any step. When mis-injection occurs during the initial calibration procedures, the laboratory is allowed to perform re-injection as long as it is within the 12-hour period.

NOTE: The steps pertaining to Instrument Blank and PEM are used as part of the continuing calibration verification as well (Section 9.4).

9.3.3.5 Choose the appropriate initial calibration sequence below (Sequence 1 or 2). If two Individual Standard Mixtures are used, choose Initial Calibration Sequence 2. The appropriate calibration sequence is determined by the results of the Resolution Check Mixture (Section 9.3.4). A single-point Toxaphene calibration at low standard shall be included in the initial calibration at a minimum. Optionally, all five-point initial calibration standards may be included in the initial calibration as in Sequence 1 or 2.

INITIAL CALIBRATION SEQUENCE 1

1. Resolution Check
2. PEM
3. Toxaphene CS1
4. Toxaphene CS2
5. Toxaphene CS3
6. Toxaphene CS4
7. Toxaphene CS5
8. CS1 Individual Standard Mixture C
9. CS2 Individual Standard Mixture C
10. CS3 Individual Standard Mixture C
11. CS4 Individual Standard Mixture C
12. CS5 Individual Standard Mixture C
13. Instrument Blank
14. PEM

INITIAL CALIBRATION SEQUENCE 2

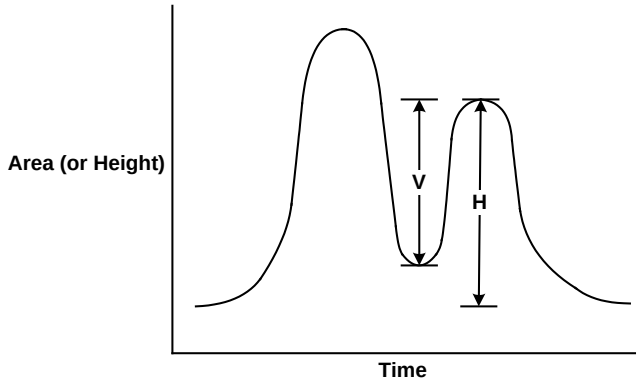
1. Resolution Check
2. PEM
3. Toxaphene CS1
4. Toxaphene CS2
5. Toxaphene CS3
6. Toxaphene CS4

7. Toxaphene CS5
8. CS1 Individual Standard Mixture A
9. CS1 Individual Standard Mixture B
10. CS2 Individual Standard Mixture A
11. CS2 Individual Standard Mixture B
12. CS3 Individual Standard Mixture A
13. CS3 Individual Standard Mixture B
14. CS4 Individual Standard Mixture A
15. CS4 Individual Standard Mixture B
16. CS5 Individual Standard Mixture A
17. CS5 Individual Standard Mixture B
18. Instrument Blank
19. PEM

9.3.4 Calculations for Initial Calibration

9.3.4.1 Calculate the resolution between the analytes in the Resolution Check Mixture, PEM, and CS3 Standard concentrations of the Individual Standard Mixtures using Equation 1.

Figure 1. Peak Height and Valley



EQ. 1 Percent Resolution

$$\%\text{Resolution} = \frac{V}{H} \times 100$$

WHERE,

V = Depth of the valley between the two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks

9.3.4.2 During the initial calibration sequence, mean RTs ($\overline{RT}$s) are determined for all single component pesticides, surrogates, and five major peaks of Toxaphene for both columns.

9.3.4.3 For each single component pesticide, an RT is measured in each of the five calibration standards for all Individual Standard Mixtures A and B and Individual Standard Mixture C. If Toxaphene is performed using a single-point calibration, use the RT for each peak from this standard. For Toxaphene five-point calibrations, an RT is measured in each of the five calibration

standards for the major peaks. The $\overline{RT}$ is calculated for each single component pesticide, surrogate, and Toxaphene as the average of the five values. Calculate the $\overline{RT}$ for each single component pesticide, surrogate, and Toxaphene using the following equation:

EQ. 2 Mean Retention Time

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

WHERE,

RT_i = Retention Time of analyte

n = Total number of measurements (n=5)

9.3.4.4 An RT window is calculated for each single component analyte and surrogate and for the five major peaks of Toxaphene using Table 5 – Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates. The $\overline{RT}$s for surrogates are calculated from the five analyses of the Individual Standard Mixtures. If two Individual Standard Mixtures are used, calculate the $\overline{RT}$s for the surrogates from the Individual Standard Mixture A only. Windows are centered around the $\overline{RT}$ for the analyte established during the initial calibration. Compounds are identified when peaks are observed in the RT window for the compound on both GC columns.

9.3.4.5 Calculate the Calibration Factors (CFs) for each single component pesticide and surrogates over the initial calibration range using Equation 3. The CFs for surrogates are calculated from the five analyses of the Individual Standard Mixtures. If two Individual Standard Mixtures are used, calculate the CFs for surrogates from Individual Standard Mixture A only. Either peak area or peak height may be used to calculate the CFs using Equation 6.

9.3.4.5.1 For example, it is permitted to calculate the CF for Endrin based on peak area and to calculate CF for Aldrin based on peak height. It is not permitted to calculate CFs for an analyte from both peak area and peak height. For example, it is not permitted to calculate the CFs for the CS1 Standard for Endrin using peak height and calculate the CS3 and CS5 Standard CFs for Endrin using peak area.

EQ. 3 Calibration Factor

$$CF = \frac{\text{Peak area (or peak height) of the standard}}{\text{Mass Injected (ng)}}$$

9.3.4.6 Calculate the Mean CF ($\overline{CF}$) and the Percent Relative Standard Deviation (%RSD) of the CF for each single component pesticide and surrogate over the initial calibration range using Equations 4 and 6.

EQ. 4 Mean Calibration Factor

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

WHERE,

CF_i = Calibration Factor

n = Total number of values (n=5)

9.3.4.7 The linearity of the instrument is determined by calculating a %RSD of the CFs from a five-point calibration curve for each of the single component pesticides and surrogates using Equations 5 and 6.

EQ 5. Standard Deviation of Calibration Factors

$$SD_{CF} = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{(n-1)}}$$

WHERE,

CF_i, $\overline{CF}$, n = As given in EQ. 4

EQ. 6 Percent Relative Standard Deviation of the Calibration Factors

$$\%RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

WHERE,

SD_{CF} = Standard Deviation of Calibration Factors

CF_i, $\overline{CF}$, n = As given in EQ. 4

9.3.4.8 Toxaphene shall be calibrated at a single low-point CS1 for pattern recognition. The Toxaphene standard may be analyzed before or after the analysis of the five levels of the single component pesticides standards during the initial calibration. A CF is calculated for each peak in a selected set of five major peaks for Toxaphene using Equation 3.

9.3.4.9 If Toxaphene is detected in a sample analysis following a single-point initial calibration, a separate five-point Toxaphene calibration must be prepared (Section 7.2.2.2) and analyzed, followed by a reanalysis of the sample. A CF is calculated for each peak in a selected set of five major peaks for Toxaphene using Equation 3. The $\overline{CF}$ and the %RSD of the CFs for each selected Toxaphene peak are calculated using Equations 4 and 6. When Toxaphene is detected in any sample without a valid five-point calibration during initial calibration, Toxaphene results are calculated by the single-point CFs. Subsequently, the sample must be reanalyzed following a valid five-point calibration of Toxaphene.

9.3.4.10 Calculate the Percent Breakdown (%Breakdown) of DDT, the Percent Breakdown of Endrin, and the combined breakdown of DDT and Endrin in the PEM using Equations 7, 8, 9, and 10.

EQ. 7 Amount Found

$$\text{Amount found (ng)} = \frac{\text{Peak area (or peak height) of compound in PEM}}{\overline{CF}}$$

WHERE,

$\overline{CF}$ = Mean Calibration Factor from the initial calibration (area/ng)

EQ. 8 Percent Breakdown of DDT

$$\%\text{Breakdown DDT} = \frac{\text{Amount found (ng) (DDD + DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

EQ. 9 Percent Breakdown of Endrin

$$\%\text{Breakdown Endrin} = \frac{\text{Amount found (ng) (Endrin Aldehyde + Endrin Ketone)}}{\text{Amount (ng) of Endrin injected}} \times 100$$

EQ. 10 Combined Percent Breakdown of DDT and Endrin

$$\text{Combined \%Breakdown} = \%\text{Breakdown DDT} + \%\text{Breakdown Endrin}$$

9.3.4.11 Calculate the Percent Difference (%D) between the calculated and nominal concentrations of each pesticide and surrogate in the PEM using Equations 7 and 11.

EQ. 11 Percent Difference Between the Calculated and Nominal Amount

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

WHERE,

C_{calc} = Calculated amount of each analyte from the analysis of the standard [Amount found (ng) in EQ. 7]

C_{nom} = Nominal amount of each analyte

9.3.5 Technical Acceptance Criteria for Initial Calibration

All initial calibration technical acceptance criteria apply independently to each GC column.

9.3.5.1 The initial calibration sequence must be analyzed according to the procedure and in the order listed in Section 9.3.3, at the concentrations listed in Section 7.2.2, and at the frequency listed in Section 9.3.2. The GC/ECD operating conditions optimized in Section 9.1 must be followed.

9.3.5.2 The identification of single component pesticides by GC methods is based primarily on RT data. The RT of the apex of a peak can only be verified from an on-scale chromatogram. The identification of Toxaphene by GC methods is based primarily on recognition of patterns of RTs displayed on a chromatogram.

Therefore, the following requirements apply to all data presented for single component and Toxaphene.

9.3.5.2.1 The chromatograms of the Resolution Check Mixture, the PEM, and the Individual Standard Mixtures analyzed during the initial calibration sequence must display the single component analytes present in each standard at greater than 10% of full scale, but less than 100% of full scale.

9.3.5.2.2 The chromatograms for at least one of the five analyses of each Individual Standard Mixture from the initial calibration sequence must display the single component analytes at greater than 50% of full scale, but less than 100% of full scale.

9.3.5.2.3 For all Resolution Check Mixtures, PEMs, Individual Standard Mixtures, and blanks, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

9.3.5.2.4 If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

9.3.5.3 The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 80% for all analytes for the primary column and greater than or equal to 50% for the confirmation column in order to use one Individual Standard Mixture (C). If two Individual Standard Mixtures (A and B) are to be used, then the resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60% for both GC columns.

9.3.5.4 All single component pesticides and surrogates in both analyses of the PEM must be greater than or equal to 90% resolved on each column.

9.3.5.5 The RTs of each of the single component pesticides and surrogates in both analyses of the PEM must be within the RT window determined from the five-point initial calibration in Sections 9.3.4.2 and 9.3.4.3.

9.3.5.6 If Individual Standard Mixture (C) is used, then the resolution between any two adjacent peaks in the CS3 Individual Standard Mixture C must be at least 80% for the primary column and 50% for the confirmation column. If two Individual Standard Mixtures (A and B) are used, then the resolution between any two adjacent peaks in the CS3 Individual Standard Mixtures (A and B) must be greater than or equal to 90% on both columns.

9.3.5.7 The %D between the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in both of the PEM analyses on each GC column must be in the inclusive range of ±25.0% when calculated using Equation 11.

9.3.5.8 The %Breakdown of DDT and Endrin in each of the PEM analyses must be ≤ 20.0% calculated using Equations 8 and 9. The combined %Breakdown of DDT and Endrin must be ≤ 30.0% calculated using Equation 10.

9.3.5.9 The %RSD of the CFs for each single component target analyte must be ≤ 20.0%, except alpha-BHC and delta-BHC. The %RSD of the CFs for alpha-BHC and delta-BHC must be ≤ 25.0%. The %RSD of the CFs for the two surrogates must be ≤20.0%. Up to two single component target analytes (not surrogates) per column may exceed the maximum %RSD of 20.0% (25.0% for alpha-BHC and delta-BHC), but those analytes must have a %RSD of less than or equal to 30.0%. The %RSD of the CFs for Toxaphene five-point calibration must be less than or equal to 30.0%.

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, reinject the initial calibration standards in sequence. If the technical acceptance criteria for the initial calibration are still not met, inspect the system for problems. It may be necessary to change the column, bake-out the detector, clean the injection port, or take other corrective actions to achieve the technical acceptance criteria.

9.3.6.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. It is recommended to refer to manufacturer's guidelines for performing detector maintenance. In the case of severe contamination, the detector may require servicing by the ECD manufacturer.

CAUTION: DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

9.3.6.3 After major maintenance is completed, the detector must be recalibrated using the initial calibration sequence.

9.3.6.4 Any samples or required blanks analyzed when the initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.4 Continuing Calibration Verification

9.4.1 Summary of Continuing Calibration Verification

Three types of analyses are used to verify the calibration and evaluate instrument performance: instrument blanks, PEMs, and the CS3 Standards. A calibration verification consists of an instrument blank and PEM, or an instrument blank and the CS3 Individual Standard Mixture(s), and a CS3 Toxaphene Standard (if necessary). Sample (including LCS and MS/MSD) and required blank (method/sulfur cleanup) data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEMs, and CS3 Standards. When Toxaphene is detected in sample analyses during the analytical sequence that includes a five-point Toxaphene calibration, the closing CCV must include the CS3 Toxaphene Standard.

9.4.2 Frequency of Continuing Calibration Verification

9.4.2.1 An instrument blank and the PEM must bracket one end of a 12-hour period during which sample and required blank data are collected, and a second instrument blank and the CS3 Individual Standard Mixture(s) must bracket the other end of the 12-hour period. If Individual Standard Mixtures A and B were used in the associated initial calibration sequence, then CS3 Individual Standard Mixtures A and B must be used for the calibration verification. If Individual Standard Mixture C was used in the associated initial calibration sequence, then CS3 Individual Standard Mixture C must be used in the calibration verification.

9.4.2.2 For the 12-hour period immediately following the initial calibration sequence, the instrument blank and the PEM that are the last two steps in the initial calibration sequence that bracket the front end of that 12-hour period. The injection of the instrument blank starts the beginning of the 12-hour period (Section 9.3.3.4). Samples (including LCSs and MS/MSDs) and required blanks (method/sulfur cleanup) may be injected during the 12 hours from the injection of the instrument blank. The first injections immediately after that 12-hour period must be an instrument blank and the CS3 Individual Standard Mixture(s). The instrument blank must be analyzed first, before the standard(s). If two Individual Standard Mixtures are used, they may be analyzed in either order (A, B or B, A).

9.4.2.3 The analyses of the instrument blank and CS3 Individual Standard Mixture(s) immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the technical acceptance criteria in Section 9.4.5. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and a PEM, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks, PEMs, or Individual Standard Mixture(s) fails to meet the technical acceptance criteria in Section 9.4.5. The 12-hour period begins with the injection of the instrument blank.

9.4.2.4 If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an acceptable instrument blank and PEM must be analyzed to start a new sequence. This requirement applies even if no analyses were performed since that standard was injected.

9.4.2.5 If the entire 12-hour period is not required for the analyses of all samples and blanks to be reported and all data collection is to be stopped, the sequence must be ended with either the instrument blank/PEM combination or the instrument blank/CS3 Individual Standard Mixture(s) combination, whichever was due to be performed at the end of the 12-hour period. For Toxaphene analyses under a five-point calibration, the sequence must end with an instrument blank and a CS3 Toxaphene Standard.

9.4.2.6 No more than 14 hours may elapse from the injection beginning the opening CCV and the injection ending the closing CCV (PEM or CS3 Standard Mixture).

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Time	Injection #	Material Injected
0 hr		Instrument Blank at end of initial calibration
		PEM at end of initial calibration
		First sample if using initial calibration
		Subsequent samples
		Last Sample

Time	Injection #	Material Injected
12 hrs	1st injection past 12 hours	Instrument blank
	Next injections past 12 hours	Individual Standard Mixtures A and B or **Individual Standard Mixture C**
		Sample
		Subsequent samples
		Last Sample
Another 12 hrs	1st injection past 12 hours	Instrument blank
	Next injection past 12 hours	PEM
		Sample
		Samples with Toxaphene detected
		Subsequent samples
		Last Sample
Another 12 hrs	1st injection past 12 hours	Instrument blank
	Next injections past 12 hours	Individual Standard Mixtures A and B or **Individual Standard Mixture C**
	Next injection past 12 hours	Toxaphene CS3

9.4.3 Procedure for Continuing Calibration Verification

9.4.3.1 All standards and instrument blanks must be allowed to warm to ambient temperature before analysis.

9.4.3.2 Analyze the instrument blank, PEM, and the CS3 Individual Standard Mixture(s) according to Section 10.4.2 using the same injection volumes as in the initial calibration.

9.4.4 Calculations for Continuing Calibration Verification

9.4.4.1 For each analysis of the PEM used to demonstrate calibration verification, calculate the %D between the amount of each analyte (including the surrogates) found in the PEM and the nominal amount, using Equation 11.

9.4.4.2 For each analysis of the PEM used to demonstrate calibration verification, calculate the %Breakdown of Endrin and DDT, and the combined %Breakdown, using Equations 7, 8, 9, and 10.

9.4.4.3 For each analysis of the CS3 Individual Standard Mixture(s) or CS3 Toxaphene used to demonstrate calibration verification, calculate the %D between the CF of each analyte (including the surrogates) in the standard mixture and the corresponding $\overline{CF}$ from the initial calibration, using Equation 12. Do not calculate the breakdown of Endrin and DDT in the Individual Standard Mixtures, as these standards contain the breakdown products as well as the parent compounds.

EQ. 12 External Standard Calibration Percent Difference

$$\%D = \frac{CF-\overline{CF}}{\overline{CF}} \times 100$$

WHERE,

CF = Calibration Factor for CS3 Standard used for Calibration Verification

$\overline{CF}$ = Mean Calibration Factor

9.4.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.4.5.1 All CCV technical acceptance criteria apply independently to each GC column, and must meet the chromatographic criteria specified in Section 9.3.5.2.

9.4.5.2 The PEMs, CS3 Standards, and instrument blanks must be analyzed at the required frequency on a GC/ECD system that has met the initial calibration technical acceptance criteria.

9.4.5.3 All single component pesticides and surrogates in the PEMs used to demonstrate calibration verification must be greater than or equal to 90.0% resolved. If one Individual Standard Mixture is used, the resolution between any two adjacent peaks in the CS3 Individual Standard Mixture C must be at least 80% for the primary column and 50% for the confirmation column. If two Individual Standard Mixtures are used, the resolution between any two adjacent peaks in the CS3 Individual Standard Mixture A and B used to demonstrate calibration verification must be greater than or equal to 90.0% for both columns.

9.4.5.4 The RT for each of the single component pesticides and surrogates in the PEMs and CS3 Standards used to demonstrate calibration verification must be within the RT windows determined from the five-point initial calibration in Sections 9.3.4.2 and 9.3.4.3.

9.4.5.5 The %D between the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM used to demonstrate calibration verification must not exceed ±25.0%.

9.4.5.6 The %Breakdown of 4,4'-DDT in the PEM must be less than or equal to 20.0% on each column. The %Breakdown of Endrin in the PEM must be less than or equal to 20.0% on each column. The combined %Breakdown of DDT and Endrin must be less than or equal to 30.0% on each column.

9.4.5.7 The %D between the CF of each of the single component pesticides and surrogates in the mid-point concentration of the Individual Standard Mixtures CS3 and the $\overline{CF}$ from the initial calibration must be in the inclusive range of ±25.0% and ±30.0%, respectively.

9.4.5.8 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.5.

9.4.5.9 A Toxaphene closing calibration verification standard (CS3) must be analyzed within a valid 12-hour analytical sequence including the reanalysis of samples in which Toxaphene was detected. The %D between the CF of each peak used to identify Toxaphene in the calibration verification standard and the $\overline{CF}$ from the initial calibration must not exceed ±25.0%.

9.4.6 Corrective Action for Continuing Calibration Verification

9.4.6.1 If the technical acceptance criteria for the CCV are not met, inspect the system for problems and take corrective action to achieve the technical acceptance criteria.

9.4.6.2 Major corrective actions, such as replacing the GC column or baking out the detector, will require that a new initial calibration be performed that meets the technical acceptance criteria in Section 9.3.5.

9.4.6.3 Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the standard (PEM or CS3 Standard) that originally failed the criteria and an associated instrument blank immediately after the corrective action do meet all the technical acceptance criteria.

9.4.6.4 If a PEM or CS3 Standard does not meet the technical acceptance criteria listed in Section 9.4.5, it must be re-injected immediately. If the second injection of the PEM or CS3 Standard meets the criteria, sample analysis may continue. If the second injection does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken and a new initial calibration sequence must be established before more sample data are collected.

9.4.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Section 12.1.4.5, all data collection must be stopped. Appropriate corrective action must be taken to clean out the system and an acceptable instrument blank must be analyzed before more sample data are collected.

9.4.6.6 The Contractor is reminded that analyzing an instrument blank and a PEM or CS3 Standard once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to analyze instrument blanks and standards more often to avoid discarding data.

9.4.6.7 If a successful instrument blank and PEM cannot be analyzed after an interruption in analysis (Section 9.4.2.6), an acceptable initial calibration must be established before sample data may be collected. All acceptable sample analyses (including LCSs and MS/MSDs) and required blank (method/sulfur cleanup) analyses must be preceded and followed by acceptable instrument blanks and standards as described in Section 9.4.2.

9.4.6.8 Any samples and required blanks associated with a CCV that do not meet the technical acceptance criteria will require reanalysis at no additional cost to the EPA.

9.4.6.9 The corrective action for sample reanalysis is not required when the noncompliant analytes or surrogates, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or surrogates for which the dilution, re-extraction, or reanalysis was intended.

10.0 PROCEDURE

The Contractor must have the capability to perform all sample cleanup procedures presented in this Exhibit. The Contractor may use any of the procedures or combinations of procedures to clean up the samples prior to analysis, unless the Contractor is specifically directed by the EPA Region to use a particular cleanup procedure or combination of cleanup procedures.

The Contractor must demonstrate that each cleanup procedure is capable of producing data that meets the technical acceptance criteria for the method, including MDLs (Section 12.4) and any precision and recovery limits.

10.1 Sample Preparation

10.1.1 Water and Leachate Samples

Water and leachate samples may be extracted by either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed. Allow the samples to warm to ambient temperature before extraction.

10.1.1.1 Separatory Funnel Extraction

10.1.1.1.1 For samples received in 1 L bottles, the Contractor shall mark the meniscus and transfer the entire sample into the separatory funnel. If the sample was not received in a 1 L bottle, measure out each 1.0 L sample aliquot in a separate graduated cylinder.

10.1.1.1.2 Measure and record the volume of sample contained in the 1 L sample bottle with water using a graduated cylinder.

10.1.1.1.3 Using a syringe or a volumetric pipette, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.2.7) to all water samples.

10.1.1.1.4 Measure and record the pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG Narrative. Place the sample aliquot into a 2 L separatory funnel.

10.1.1.1.5 Rinse the 1 L sample bottle and/or graduated cylinder with 30 mL of methylene chloride and transfer the rinsate to the separatory funnel.

10.1.1.1.6 Add another 30 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for 2 minutes, with periodic venting to release excess pressure.

NOTE: The total volume of solvent used for extraction is 60 mL. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than 1/3 the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration through glass wool, centrifugation, or other physical means. Drain the methylene chloride into a 250 mL Erlenmeyer flask.

10.1.1.1.7 Add a second 60 mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Proceed to Section 10.2.

10.1.1.2 Continuous Liquid-Liquid Extraction

10.1.1.2.1 Continuous Liquid-Liquid Extraction without Hydrophobic Membrane

10.1.1.2.1.1 Follow the manufacturer's instructions for set-up.

10.1.1.2.1.2 Add 300-500 mL of methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom sidearm.

10.1.1.2.1.3 If the samples have been received in 1 L bottles, the Contractor shall mark the meniscus and transfer the entire sample into the continuous extractor. If the sample was not received in a 1 L bottle, measure out each 1.0 L sample aliquot in a separate, clean graduated cylinder and transfer the aliquot to the continuous extractor.

10.1.1.2.1.4 Using a syringe or volumetric pipette, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.2.7) into the sample and mix well. Perform spiking prior to pH adjustment or any other processing steps.

10.1.1.2.1.5 Measure the pH of the sample with wide range pH paper or a pH meter and record the pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring the pH adjustment must be noted in the SDG Narrative.

NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.1.2.1.6 Rinse the graduated cylinder with a small amount of methylene chloride and transfer the rinsate to the continuous extractor. If the sample container is empty, rinse the container with a small amount of methylene chloride and add the rinsate to the continuous extractor.

10.1.1.2.1.7 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5-15 mL/minute (recommended); optimize the extraction drip rate. Extract for a <u>minimum</u> of 18 hours.

NOTE 1: When a minimum drip rate of 10-15 mL/minute is maintained throughout the extraction, the extraction time may be reduced to a <u>minimum</u> of 12 hours. Allow to cool, and then detach the distillation flask. Proceed to Section 10.2.

NOTE 2: Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.1.2.2 Continuous Liquid-Liquid Extraction with Hydrophobic Membrane

10.1.1.2.2.1 Follow the procedure in Sections 10.1.1.2.1.1 - 10.1.1.2.1.5, but reduce the amount of methylene chloride used to 50 mL and extract for a minimum of 6 hours.

10.1.1.2.2.2 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours.

10.1.1.2.2.3 Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion that will consume the solvent volume, preventing efficient extraction of the sample. When this occurs, add additional solvent to ensure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used. Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

10.1.1.2.2.4 Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

10.1.1.2.2.5 If low surrogate recoveries occur, ensure that: 1) the apparatus was properly assembled to prevent leaks, 2) the drip rate/solvent cycling was optimized, and 3) there was proper cooling for condensation of solvent. Document the problem and the corrective action.

10.1.1.2.2.6 Alternate continuous extractor types that meet the requirements of the analytical method may also be used. If using alternate extractors or design types, follow the manufacturer's instructions for set-up. Optimize the extraction procedure.

10.1.2 Soil/Sediment Samples

Mix samples thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase.

10.1.2.1 Extraction of Soil/Sediment Samples

10.1.2.1.1 Three procedures are provided for the extraction of pesticide analytes from soil/sediment samples:

- ultrasonic extraction;
- Soxhlet extraction (automated and manual); and
- pressurized fluid extraction (PFE).

NOTE: All soil/sediment samples in a Case must be extracted by the same procedure.

10.1.2.1.2 For soil/sediment sample extractions, weigh 30-50 g of sample, to the nearest 0.1 g, into a 400 mL beaker. 30 g is ideal, as more sample may be used to compensate for high moisture content. If the system cannot accommodate 30 g of a sample, a smaller sample size may be used. The specified CRQLs must be met. Adjust the amount of solvents and standards added as necessary. Document the smaller sample size in the SDG Narrative along with all steps taken to ensure sample homogeneity.

10.1.2.1.3 Add 60 g of anhydrous powdered or granulated sodium sulfate, or add 30 g of Hydromatrix™, and mix well to produce a sandy texture. Additional drying agent may be added as needed.

NOTE: For samples extracted by the PFE procedure (Section 10.1.2.1.7) the use of sodium sulfate is not recommended.

10.1.2.1.4 Add 1.0 mL of surrogate standard spiking solution (Section 7.2.2.7) to the sample, then immediately add 100 mL of 1:1 (v/v) acetone/methylene chloride. Proceed to Section 10.1.2.1.5 for ultrasonic extraction, Section 10.1.2.1.6 for automated Soxhlet extraction, or Section 10.1.2.1.7 for pressurized fluid extraction. As applicable, follow the manufacturer's instructions for use of all extraction equipment.

10.1.2.1.5 Ultrasonic Extraction

10.1.2.1.5.1 Place the bottom surface of the tip of the 3/4-inch tapered disruptor horn about 1/2 inch below the surface of the solvent, but above the sediment layer. Do <u>not</u> use a microtip probe.

10.1.2.1.5.2 Sonicate for 3 minutes with output at full power with pulse on (pulsing energy as opposed to continuous), and percent duty cycle knob set at 50%.

NOTE: Refer to the manufacturer's instructions for appropriate output settings.

10.1.2.1.5.3 Transfer and filter extracts through Whatman No. 42 (or equivalent) filter paper using vacuum filtration or centrifuge and decant extraction solvent.

10.1.2.1.5.4 Repeat the extraction two more times with two additional 100 mL portions of 1:1 (v/v) acetone/methylene chloride. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with a clean spatula. Transfer the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 (v/v) acetone/methylene chloride. Proceed to Section 10.2.

10.1.2.1.6 [Automated] Soxhlet Extraction

The Contractor may use either automated or non-automated Soxhlet extraction. The following procedure is based on the use of a Soxtec HT-6 automated Soxhlet extraction system. When using a different system, refer to the instructions provided by the manufacturer for the appropriate procedure.

10.1.2.1.6.1 Check the heating oil level in the automated Soxhlet unit and add oil if needed. Follow the manufacturer's instructions to set the temperature on the service unit.

10.1.2.1.6.2 Press the "MAINS" button and observe that the switch lamp is now "ON". Open the cold water tap for the reflux condensers. Adjust the flow to 2 L/minute to prevent solvent loss through the condensers.

10.1.2.1.6.3 Transfer the entire sample from the beaker (Section 10.1.2.1.4) to the thimble.

10.1.2.1.6.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

10.1.2.1.6.5 Insert the extraction cups containing boiling chips, and load each with an appropriate volume of 1:1 (v/v) acetone/methylene chloride. Using the cup holder, lower the locking handle and ensure that the safety catch engages. The cups are now clamped into position.

NOTE: The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.

10.1.2.1.6.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

10.1.2.1.6.7 Move the extraction knobs to the "RINSING" position. The thimbles will now hang above the solvent surface. Set timer for 60 minutes. Condenser valves are still open. Extract for the preset time. After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise.

10.1.2.1.6.8 When all but 2-5 mL of the solvent have been collected, open the system and remove the cups. Transfer the contents of the cups to graduated, conical-bottom glass tubes. Rinse the cups with methylene chloride and add the rinsates to the glass tubes. Proceed to Section 10.2.

10.1.2.1.7 Pressurized Fluid Extraction

10.1.2.1.7.1 Transfer the entire sample from the beaker (Section 10.1.2.1.4) to an extraction cell of the appropriate size for the aliquot.

10.1.2.1.7.2 Place the extraction cell into the instrument or autosampler tray, as described by the instrument manufacturer.

10.1.2.1.7.3 Place a pre-cleaned collection vessel in the instrument for each sample, as described by the instrument manufacturer. The total volume of the collected extract will depend on the specific instrumentation and the extraction procedure recommended by the manufacturer and may range from 0.5-1.4 times the volume of the extraction cell. Ensure that the collection vessel is sufficiently large to hold the extract.

10.1.2.1.7.4 The following are recommended extraction conditions:

Oven temperature:	100°C
Pressure:	1500-2000 psi
Static time:	5 min. (after 5 min. pre-heat equilibration)
Flush volume:	60% of the cell volume
Nitrogen purge:	60 sec. at 150 psi (purge time may be extended for larger cells)
Static cycles:	1

10.1.2.1.7.5 Optimize the extraction conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500-2000 psi should suffice. An appropriate amount of 1:1 (v/v) acetone/methylene chloride should be used to achieve the conditions in Section 10.1.2.1.7.4.

10.1.2.1.7.6 Once established, the same pressure should be used for all samples in the same SDG.

10.1.2.1.7.7 Begin the extraction according to the manufacturer's instructions. Collect each extract in a clean vial. Allow the extracts to cool after the extractions are complete. Proceed to Section 10.2.

10.2 Extract Concentration

10.2.1 Concentration by Kuderna-Danish

10.2.1.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D concentrator, if equivalency is demonstrated for all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

10.2.1.2 For water samples, transfer the extract to a K-D concentrator by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate.

10.2.1.3 For soil/sediment samples, directly transfer the extract to the K-D concentrator, if the extract is known to be dry.

10.2.1.4 Rinse the original container collecting the extract (for both water and soil/sediment samples) and the column (for water samples) with at least two 20-30 mL portions of methylene chloride to complete the quantitative transfer.

10.2.1.5 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-70°C recommended) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-30 minutes. At the proper rate of

distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 3-5 mL for water samples (and less than 10 mL for soil/sediment samples), remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.2.1.6 For water extracts that do not require GPC cleanup, and for water and soil/sediment extracts that have been through the GPC cleanup step, proceed with the hexane exchange described in Section 10.2.2.

10.2.1.7 For water extracts that require GPC cleanup, remove the Snyder column, rinse the flask and its lower joint, collect the rinsate in the concentrator tube, and adjust the volume to 10 mL with methylene chloride. Proceed to Section 10.3.1.

10.2.1.8 For soil/sediment extracts that have not been cleaned-up using GPC, it is absolutely necessary to further reduce the volume of all soil/sediment extracts to 1 mL in order to remove most of the acetone. This is best accomplished using the nitrogen evaporation technique (Section 10.2.3.2). The presence of acetone will cause a dead volume to develop in the GPC column and thus will cause loss of surrogates and analytes during GPC cleanups. Adjust the soil/sediment extract volume to 10 mL with methylene chloride. Proceed to Section 10.3.1 for mandatory GPC.

10.2.2 Solvent Exchange into Hexane

This procedure applies to both extracts of water samples and extracts of soil/sediment samples.

10.2.2.1 With the extract in a K-D apparatus, remove the Snyder column, add 50 mL of hexane and a new boiling chip, and re-attach the Snyder column. Pre-wet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as described previously (Section 10.2.1), but increase the temperature of the water bath (80-90°C recommended) to maintain proper distillation. When the apparent volume of liquid reaches 3-5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.2.2.2 Remove the Snyder column. Using 1-2 mL of hexane, rinse the flask and its lower joint into the concentrator tube. Complete quantitative transfer of the extract to a 10 mL vial by using hexane.

10.2.2.3 For samples that have <u>not</u> been subjected to GPC cleanup, adjust the volume of the hexane extract to 10 mL. For samples that <u>have</u> been subjected to GPC cleanup, concentrate the hexane extract to 5.0 mL using a Micro Snyder Column or nitrogen evaporation, as described in Section 10.2.3.1 or 10.2.3.2, then proceed to Section 10.3.2 for Florisil cartridge cleanup.

10.2.3 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to volume before Florisil cleanup or instrumental analysis. They are the Micro Snyder Column and the Nitrogen Evaporation Technique.

10.2.3.1 Micro Snyder Column Concentration

10.2.3.1.1 Add another one or two clean boiling chips to the concentrator tube and attach a two-ball Micro Snyder Column. Pre-wet the Snyder column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath

(80-90°C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and lower joint into the concentrator tube with 0.2 mL of hexane.

10.2.3.1.2 If GPC cleanup is needed and not yet performed, adjust the volume to 10 mL with methylene chloride and proceed to Section 10.3.1 for GPC cleanup. For water samples that do not require GPC cleanup, adjust the volume to 10 mL with hexane and proceed to Section 10.3.2 for Florisil cleanup. For soil/sediment samples that have already undergone GPC cleanup, adjust the volume with hexane to 5.0 mL and proceed to Section 10.3.2 for Florisil cleanup. If no further cleanup is needed, adjust the volume with hexane to the same volume of the aliquot used for Florisil and/or sulfur cleanup (1 or 2 mL) and proceed to Section 10.4 for GC/ECD analysis. Extracts may be stored at ≤6°C, but not frozen, prior to analysis.

10.2.3.2 Nitrogen Evaporation Technique

10.2.3.2.1 Place the concentrator tube in a warm water bath (30-35°C recommended) and evaporate the solvent volume to the final volume using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). DO NOT ALLOW THE EXTRACT TO GO DRY.

10.2.3.2.2 If GPC cleanup is needed and not yet performed, adjust the volume to 10 mL with methylene chloride and proceed to Section 10.3.1 for GPC cleanup. For water samples that do not require GPC cleanup, adjust the volume to 10 mL with hexane and proceed to Section 10.3.2 for Florisil cleanup. For soil/sediment samples that have already undergone GPC cleanup, adjust the volume with hexane to 5.0 mL and proceed to Section 10.3.2 for Florisil cleanup. If no further clean-up is needed, adjust the volume with hexane to the same volume of the aliquot used for Florisil and/or sulfur clean-up (1.0 or 2.0 mL) and proceed to Section 10.4 for GC/ECD analysis. Extracts may be stored at ≤6°C, but not frozen, prior to analysis.

10.2.3.2.3 Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or PTFE tubing. Plastic tubing must not be used between the carbon trap and the sample, as it may introduce interferences. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use.

10.3 Cleanup Procedures

There are three cleanup procedures specified in this method: GPC cleanup, Florisil cartridge cleanup, and sulfur cleanup. GPC cleanup must be performed for all soil/sediment extracts. GPC cleanup may be performed for water extracts that contain higher molecular weight contaminants that interfere with the analysis of the target analytes. Florisil cartridge cleanup is mandatory for all extracts. Sulfur cleanup must be performed for all sample extracts contaminated with

sulfur. Method blanks must be subjected to the same cleanup procedures as the samples (including LCSs and MS/MSDs).

10.3.1 Gel Permeation Chromatography

10.3.1.1 Introduction

GPC is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the size of the molecules to be separated.

10.3.1.2 GPC Column Preparation

Prepare the GPC column using Bio Beads. Alternate column packings may be used if: 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration verification, and 2) the column packings do not introduce contaminants/artifacts into the sample that interfere with the analysis of the pesticide analytes. Follow the manufacturer's instructions for preparation of the GPC column.

10.3.1.3 Calibration of GPC

10.3.1.3.1 Summary of GPC Calibration

The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column.

10.3.1.3.2 Frequency of GPC Calibration

Each GPC system must be calibrated prior to processing samples under the contract, when the GPC calibration verification solution fails to meet criteria (Section 10.3.1.3.4), when the column is changed, when channeling occurs, and once every 7 days when in use. Also, the RT shift must be less than 5% when compared to RTs in the last calibration UV traces.

10.3.1.3.3 Procedure for GPC Calibration

Follow the manufacturer's instructions for operating the GPC system. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte RTs and must be monitored.

10.3.1.3.3.1 Using a 10 mL syringe, load the calibration solution (Section 7.2.2.5.1) onto the GPC. Determine the elution times for bis(2-ethylhexyl)phthalate, methoxychlor, and perylene. Bis(2-ethylhexyl)phthalate will elute first; perylene will elute last.

10.3.1.3.3.2 Choose a "DUMP" time that removes greater than 85% of the phthalate. Choose a "COLLECT" time so that greater than 95% of the methoxychlor is collected, and continue to collect until just prior to the elution time of sulfur. Use a "WASH" time of 10 minutes.

NOTE: The "DUMP" and "COLLECT" times must be adjusted to compensate for the difference in volume of the lines between the detector and the collection flask.

10.3.1.3.3.3 Reinject the calibration solution after appropriate "COLLECT" and "DUMP" cycles have been set, and the solvent flow and column pressure have been established.

10.3.1.3.3.4 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate problems with the system during sample processing.

10.3.1.3.3.5 Analyze a GPC blank of methylene chloride. Concentrate the methylene chloride that passed through the system during the "COLLECT" cycle using a K-D evaporator. Exchange the solvent to hexane and analyze the concentrate by GC/ECD according to the usual protocol. Assuming that the blank represents the extract from a 1 L water sample, calculate the analyte concentrations using Equation 14.

10.3.1.3.4 Technical Acceptance Criteria for GPC Calibration

10.3.1.3.4.1 The GPC system must be calibrated at the frequency described in Section 10.3.1.3.2. The UV trace must meet the following requirements:

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution;
- Corn oil and phthalate peaks should exhibit greater than 85% resolution;
- Phthalate and methoxychlor peaks should exhibit greater than 85% resolution;
- Methoxychlor and perylene peaks should exhibit greater than 85% resolution; and
- Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

10.3.1.3.4.2 The solvent flow rate and column pressure must be within the manufacturer’s specified ranges.

10.3.1.3.4.3 The RTs for bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations. Excessive RT shifts are caused by the following:

- Poor laboratory temperature control or system leaks;
- An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight; and/or
- Excessive laboratory temperatures causing outgassing of the methylene chloride.

10.3.1.3.4.4 The analyte concentrations in a GPC blank must be less than the CRQL for all target analytes in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

10.3.1.3.4.5 A copy of the two most recent UV traces of the calibration solution must be submitted with the data for the associated samples.

10.3.1.3.5 Corrective Action for GPC Calibration

10.3.1.3.5.1 If the flow rate and/or column pressure do not fall within the manufacturer’s specified ranges, a new column should be prepared.

10.3.1.3.5.2 A UV trace that does not meet the criteria in Section 10.3.1.3.4 would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

10.3.1.3.5.3 If the GPC blank exceeds the requirements in Section 10.3.1.3.4.4, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

10.3.1.4 GPC Calibration Verification

10.3.1.4.1 Summary of GPC Calibration Verification

The GPC calibration must be routinely verified with the calibration verification check solution (Section 7.2.2.5.2).

10.3.1.4.2 Frequency of GPC Calibration Verification

10.3.1.4.2.1 The calibration verification must be performed at least once every 7 days (immediately following the GPC Calibration) whenever samples (including MS/MSDs and blanks) are cleaned up using the GPC.

10.3.1.4.2.2 Some samples may contaminate the SX-3 Bio Beads and change the retention volume of the GPC column. Therefore, system calibration and analyte recovery must be checked whenever a sample causes significant discoloration of the GPC column. Even if no darkening is visible, GPC calibration must be checked not less than once every 7 days.

10.3.1.4.3 Procedure for GPC Calibration Verification

The instructions below are for a GPC injection loop of 5 mL. If a 2 mL injection loop is used, the Contractor should adjust the volume to 4 mL instead of 10 mL before the injection of the extract on the GPC.

10.3.1.4.3.1 The GPC calibration verification solution contains gamma-BHC (Lindane), Heptachlor, Aldrin, and 4,4'-DDT, Endrin, and Dieldrin in methylene chloride at the concentrations in Table 7 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions.

10.3.1.4.3.2 Load the 5 mL sample loop by using a 10 mL syringe containing at least 8 mL of the GPC calibration verification solution. Fractions are collected in an auto-sequence by using the GPC program established by the UV detector calibration procedure (Section 10.3.1.3).

10.3.1.4.3.3 The collected GPC calibration verification fraction is transferred to a K-D apparatus, and the collection vessel is rinsed with two additional 10 mL portions of methylene chloride to complete the transfer. The volume of methylene chloride is reduced according to Section 10.2.1. After cooling, the solvent is exchanged to hexane according to the instructions in Section 10.2.2. The final volume is adjusted to 10 mL, and the sample is analyzed by GC according to the procedure in Section 10.4. The analysis must be performed on only one of the GC columns used for sample analysis.

10.3.1.4.3.4 The recovery of each analyte must be determined for evaluation and reporting purposes. Calculate the Percent Recovery (%R) of each analyte using Equation 13 in Section 10.3.2.2.3.

10.3.1.4.4 Technical Acceptance Criteria for GPC Calibration Verification

The recovery of each of the analytes must be between 80-110%.

10.3.1.4.5 Corrective Action for GPC Calibration Verification

The Contractor may continue to use the GPC column if the technical acceptance criteria for the GPC calibration verification are met. If the recoveries are out of the acceptance criteria, the columns must be replaced and the GPC recalibrated according to the instructions in Section 10.3.1.3 before proceeding with any GPC cleanup on samples (including LCSs and MS/MSDs) and required method blanks.

10.3.1.5 Daily Ultraviolet Calibration Check (Optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (Section 7.2.2.5.1) and the UV detector calibration procedure (Section 10.3.1.3.3). The UV detector should be used to monitor the elution times for the phthalate, methoxychlor, and perylene, in that order. The precalibrated GPC program should "DUMP" greater than 85% of the phthalate and should "COLLECT" greater than 95% of the methoxychlor and perylene. Significant changes in elution times of the analytes (e.g., greater than 30 seconds) indicate that the column is out of calibration and must be recalibrated or replaced.

10.3.1.6 Sample Extract Cleanup by GPC

10.3.1.6.1 Summary of GPC Cleanup

10.3.1.6.1.1 It is very important to have consistent laboratory temperatures during an entire GPC analysis, which could be 24 hours or more. If temperatures are not consistent, RTs will shift, and the "DUMP" and "COLLECT" times determined by the calibration standard will no longer will be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C.

10.3.1.6.1.2 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of 1:1 (v/v) glycerol/water solution must be diluted and loaded into several loops. Similarly, extracts containing more than the manufacturer recommended non-volatile residue must be diluted and loaded into several loops. The non-volatile residue may be determined by evaporating a 100 µL aliquot of the extract to dryness in a tared aluminum weighing pan, or another suitable container.

10.3.1.6.1.3 Systems using automated injection devices to load the sample on the column must be carefully monitored to assure that the required amount is injected onto the column. Viscous extracts or extracts containing large amounts of non-volatile residue will cause problems with injecting the proper amount of sample extract onto the column using automated injection systems. After the sample extract has been processed, the remaining sample extract in an injection vial must be checked to assure that the proper

amount of extract was injected on the column before proceeding with the extract cleanup. If the proper amount of extract was not injected, the sample must be reprepared at no additional cost to the EPA, and the sample extract must be either diluted and loaded into several loops, or the sample extract must be injected manually.

10.3.1.6.2 Frequency of Sample Extract Cleanup by GPC

GPC cleanup must be performed at least once for each soil/sediment extract that contains high molecular weight contaminants that interfere with the analysis of the target analytes. All associated QC samples (blanks, LCSs, and MS/MSDs) must be subjected to this procedure. GPC cleanup on the method blank must be performed after all associated samples have been cleaned up (GPC sequence: calibration, sample 1, sample 2, etc., method blank, calibration verification).

10.3.1.6.3 Procedure for Sample Extract Cleanup by GPC

10.3.1.6.3.1 Particles greater than 5 microns may scratch the valve, which may result in a system leak and cross-contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Draw the sample extract through the filter assembly and into the 10 mL syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container (e.g., a 15 mL culture tube with a PTFE-lined screw-cap).

10.3.1.6.3.2 Alternatively, draw the extract into the syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 mL of extract into a 10 mL syringe.

NOTE 1: Some GPC instrument manufacturers recommend using a smaller micron size filter disc. Follow the manufacturer’s recommended operating instructions.

NOTE 2: INTRODUCTION OF PARTICULATES OR GLASS WOOL INTO THE GPC SWITCHING VALVES MAY REQUIRE FACTORY REPAIR OF THE APPARATUS.

10.3.1.6.3.3 Follow the manufacturer’s instructions for operation of the GPC system being utilized. A 2 mL injection loop may be used in place of a 5 mL injection loop. If a 2 mL injection loop is used, concentrate the extract to 4 mL instead of 10 mL, and then inject 4 mL instead of 10 mL.

10.3.1.6.3.4 If the sample is difficult to load, some of the system may be blocked. Take appropriate corrective action, following the manufacturer’s recommendations. The problem must be resolved prior to loading sample extracts.

10.3.1.6.3.5 After loading each sample loop, wash the loading port with methylene chloride to minimize cross-contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

10.3.1.6.3.6 After loading the samples, process each sample using the "COLLECT" and "DUMP" cycle times established in Section 10.3.1.

10.3.1.6.3.7 Collect each sample in a 250 mL Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation, or directly into a K-D evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

- Change in solvent flow rate, caused by channeling in the column or changes in column pressure;
- Increase in column operating pressure due to the accumulation of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used; and/or
- Leaks in the system or significant variances in room temperature.

10.3.1.6.3.8 After the appropriate GPC fraction has been collected for each sample, concentrate the extract as per Section 10.2.1 and proceed to solvent exchange into hexane as described in Section 10.2.2 and Florisil cleanup in Section 10.3.2.

NOTE: Any samples that were loaded into multiple loops must be recombined before proceeding with concentration.

10.3.2 Florisil Cartridge

10.3.2.1 Summary of Florisil Cartridge Cleanup

Florisil cartridge cleanup significantly reduces matrix interference caused by polar compounds and is required for all extracts. The same volume of the concentrated extract taken for Florisil cleanup must be maintained after Florisil cleanup (1.0 or 2.0 mL).

10.3.2.2 Florisil Cartridge Performance Check

10.3.2.2.1 Summary of Florisil Cartridge Performance Check

Every lot number of Florisil cartridges must be tested before it is used for sample cleanup.

10.3.2.2.2 Frequency of Florisil Cartridge Performance Check

The Florisil cartridge performance check must be conducted at least once on each lot of cartridges used for sample cleanup or every 6 months, whichever is most frequent.

10.3.2.2.3 Procedure for Florisil Cartridge Performance Check

Add 0.5 mL of 2,4,5-trichlorophenol solution (0.10 µg/mL in acetone; Section 7.2.2.6) and 0.5 mL of Individual Standard Mixture A or C (mid-point concentration; Section 7.2.2.3) to 4 mL of hexane. Reduce the volume to 0.5 mL using nitrogen (Section 10.2.3.2). Place the mixture onto the top of a washed Florisil cartridge, and elute it with 9 mL of hexane/acetone [(90:10) (V/V)]. Use two additional 1 mL hexane rinses to ensure quantitative transfer of the standard from the cartridge. Concentrate to a final volume of 1 mL and analyze the solution by GC/ECD using at least one of the GC columns specified for sample analysis. Determine the recovery of each analyte for evaluation and reporting purposes. Calculate the %R using Equation 13.

EQ. 13 Percent Recovery

$$\%R = \frac{(Q_d \times DF)}{Q_a} \times 100$$

WHERE,

Q_d = Quantity determined by analysis

Q_a = Quantity added

DF = Dilution Factor

NOTE: For the Florisil cartridge performance check, use DF = 1.0 in calculations.

10.3.2.2.4 Technical Acceptance Criteria for Florisil Cartridge Performance Check

10.3.2.2.4.1 The Florisil cartridge performance check solution must be analyzed on a GC/ECD meeting the initial calibration and CCV technical acceptance criteria.

10.3.2.2.4.2 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80-120% (Table 8 – Florisil Cartridge Performance Check), if the recovery of 2,4,5-trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

10.3.2.2.5 Corrective Action for Florisil Cartridge Performance Check

Any lot of Florisil cartridges that does not meet the criteria above must be discarded and a new lot, meeting criteria, must be used for sample cleanup.

10.3.2.3 Sample Extract Cleanup by Florisil Cartridge

10.3.2.3.1 Summary of Florisil Cartridge Cleanup

The required Florisil cartridge size and the final volume of the extract after Florisil cleanup are a function of the GC autosampler that a laboratory uses. If the autosampler operates reliably with 1.0 mL of sample extract, then a 500 mg cartridge is used and the required final volume is 1 mL. If the autosampler requires more sample, prepare 2 mL of sample extract using a 1 g cartridge. Manual injection requires only a 1 mL final extract and a 500 mg cartridge.

10.3.2.3.2 Frequency of Sample Extract Cleanup by Florisil Cartridge

All sample extracts (including LCSs and MS/MSDs) and method blank extracts are required to be cleaned up by the Florisil cartridge technique.

10.3.2.3.3 Procedure for Sample Extract Cleanup by Florisil Cartridge

10.3.2.3.3.1 Attach the vacuum manifold to a water aspirator or to a vacuum pump with a trap installed between the manifold and the vacuum source. Adjust the vacuum pressure in the manifold to between 5-10 lbs of vacuum.

10.3.2.3.3.2 Place one Florisil cartridge into the vacuum manifold for each sample extract.

10.3.2.3.3.3 Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge on the vacuum manifold, by pulling a vacuum, and by passing at least 5 mL of the hexane/acetone solution through the cartridge. While the cartridges are

being washed, adjust the vacuum applied to each cartridge so that the flow rate through each cartridge is approximately equal. DO NOT ALLOW THE CARTRIDGES TO GO DRY AFTER THEY HAVE BEEN WASHED.

10.3.2.3.3.4 After the cartridges on the manifold are washed, the vacuum is released, and a rack containing labeled 10 mL volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside of the appropriate volumetric flask as the manifold top is replaced.

10.3.2.3.3.5 After the volumetric flasks are in place, the vacuum to the manifold is restored, and a volume of extract equal to the required final volume (1.0 or 2.0 mL) from each sample, blank, or Matrix Spike extract is transferred to the top frit of the appropriate Florisil cartridge. This must equal the final volume after Florisil cleanup.

10.3.2.3.3.6 Because the volumes marked on concentrator tubes are not necessarily accurate at the 1 mL level, the use of a syringe or a volumetric pipette is required to transfer the extract to the cleanup cartridge.

10.3.2.3.3.7 The pesticides in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and collected into the 10 mL volumetric flasks held in the rack inside the vacuum manifold.

10.3.2.3.3.8 Transfer the eluate in each volumetric flask to a clean centrifuge tube or 10 mL vial. Use two additional 1 mL hexane rinses to ensure quantitative transfer of the cartridge eluate.

10.3.2.3.3.9 Adjust the extract to the same 1.0 or 2.0 mL aliquot volume as was taken for cleanup using either of the blowdown techniques (Section 10.2.3.1 or 10.2.3.2). Measure the final volume with a syringe or by transferring the extract to a volumetric flask.

10.3.2.3.3.10 If sulfur cleanup is to be performed, proceed to Section 10.3.3. Otherwise, transfer the sample to a GC vial and label the vial. The extract is ready for GC/ECD analysis.

10.3.3 Sulfur Cleanup

10.3.3.1 Summary of Sulfur Cleanup

Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analyses of the later eluting pesticides. If crystals of sulfur are evident or if the presence of sulfur is suspected, sulfur removal must be performed. Interference which is due to sulfur is not acceptable. Sulfur can be removed by one of two methods, according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract, and withdraw the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

10.3.3.2 Frequency of Sulfur Cleanup

Sulfur removal is required for all sample extracts that contain sulfur.

10.3.3.3 Procedure for Sulfur Cleanup

10.3.3.3.1 Removal of Sulfur using Tetrabutylammonium (TBA) Sulfite

The TBA Sulfite procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

Add 2 mL TBA Sulfite Reagent, 1 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed, add one or two more aliquots (approximately 0.65 g) to the extract and observe. Place the samples on the wrist shaker for 45 minutes, observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5.0 mL organic free water and shake for 10-15 minutes. Place the samples into the centrifuge and spin at a setting and duration appropriate to spin down the solids. Transfer the hexane layer to a clean 10 mL vial and cap. The extract transferred to the vial still represents the 1.0 or 2.0 mL final volume.

10.3.3.3.2 Removal of Sulfur using Copper

Add approximately 2 g of cleaned copper powder to the extract in the centrifuge or concentrator tube (2 g will fill the tube to about the 0.5 mL mark). Mix the copper and extract for at least 1 minute on a mechanical shaker. Separate the extract from the copper powder by drawing off the extract with a disposable pipette, and transfer the extract to a clean vial. The extract transferred to the vial still represents the 1 or 2 mL of extract. The separation of the extract from the copper powder is necessary to prevent degradation of the pesticides. If the copper appears bright, proceed to Section 10.4 and analyze the extract. If the copper changes color, repeat the sulfur removal procedure as necessary.

10.4 Gas Chromatography/Electron Capture Detector Analysis

10.4.1 Introduction

10.4.1.1 Before samples (including LCSs and MS/MSDs) and required blanks (method, sulfur cleanup, and/or instrument) can be analyzed, the instrument must meet the initial calibration and CCV technical acceptance criteria. All sample extracts, required blanks, and calibration standards must be analyzed under the same instrumental conditions. All sample extracts, required blank extracts, and standard/spiking solutions must be allowed to warm to ambient temperature before preparation/analysis. Sample analysis on two different non-equivalent GC columns (Section 6.3.2) is required for all samples and blanks.

10.4.1.2 Set up the GC/ECD system per the requirements in Section 9.1. Unless ambient temperature on-column injection is used, the injector must be heated to at least 200°C. The optimized GC conditions must be used.

10.4.2 Procedure for Sample Analysis by GC/ECD

The injection must be made on-column by using either automatic or manual injection. 1.0 or 2.0 µL injector volumes may be used provided that all associated standards, samples, and blanks use the same injection volume. The same injection volume must be used for all standards, samples (including LCSs and MS/MSDs), and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2.0 µL. However, the same injection volume must be used for all analyses.

10.4.2.1 Analytical Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below.

NOTE: The injection # will depend on whether initial calibration sequence 1 or 2 is used.

Time	**Injection #**	**Material Injected**
	12 steps (sequence 1) or 17 steps (sequence 2)	First steps of the initial calibration sequence 1 or 2
0 hr	1st injection past the Initial Calibration sequence	Instrument Blank at end of initial calibration sequence
	2nd injection past the Initial Calibration sequence	PEM at end of initial calibration sequence
		First sample following initial calibration sequence
		Subsequent samples
		Last Sample
12 hrs	1st injection past 12 hours	Instrument Blank
	2nd and 3rd injections past 12 hours	Individual Standard Mixtures A and B
	2nd injection past 12 hours	Individual Standard Mixture C
		Sample
		Subsequent samples
		Last Sample
Another 12 hrs	1st injection past 12 hours	Instrument Blank
	2nd injection past 12 hours	PEM
		Sample
	2nd last injection of 12 hours	Instrument Blank
	Last injection of 12 hours	CCV

10.4.2.1.1 For initial calibration sequence 2, the first 12 hours are counted from injection #18 (the Instrument Blank at the end of the initial calibration sequence), not from injection #1. Samples and required blanks may be injected until 12 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that brackets the front end of the samples. If more than 12 hours elapse between the injection of two instrument blanks that bracket a 12-hour period in which samples or required blanks are analyzed, then the time between the injection of the second instrument blank and the preceding sample may not exceed the length of one chromatographic run. While the 12-hour period may not be exceeded, the laboratory may analyze instrument blanks and standards more frequently, for instance, to accommodate staff working on 8-hour shifts. No more than 14 hours may elapse from the injection beginning the opening CCV (instrument blank) and the injection ending the closing CCV (PEM or Individual Standard Mixture).

10.4.2.1.2 After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks, PEMs, and Individual Standard Mixtures (A and B) or C are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be analyzed at the discretion of the Contractor; however, the blanks and standards must also satisfy the criteria presented in Sections 12.0 and 9.0 in order to continue the analytical sequence.

10.4.2.1.3 An analysis sequence must also include all samples and required blank analyses, but the Contractor may decide at what point in the sequence they are to be analyzed.

10.4.2.1.4 The requirements for the analysis sequence apply to both GC columns and for all instruments used for these analyses.

10.4.3 Sample Dilutions

10.4.3.1 All samples must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography as defined in Section 11.3.

10.4.3.2 Use the results of the original analysis to determine the approximate DF required to get the largest analyte peak (for the lower of the two column concentrations) within the initial calibration range.

10.4.3.3 If more than two analyses (i.e., from the original sample extract and more than one dilution, or from the most concentrated dilution analyzed and further dilutions) are required to get all target analytes within the calibration range, contact the Sample Management Office (SMO).

10.4.3.4 If the concentration of any single component pesticide is greater than the concentration of the high standard (CS5) of the initial calibration range on both GC columns, then the extract must be diluted. The concentration of the pesticide analyte(s) in the diluted extract must be between the initial calibration low-point (CS1) and high-point (CS5) standards for the lower column concentration of the two analyses.

10.4.3.5 If the concentration of any Toxaphene peak used for quantitation is greater than the concentration of the corresponding Toxaphene peak in the high standard (CS5) on both columns, then the sample must be diluted to have the concentration of the same peak be between the mid-point (CS3) and high-point (CS5) standards of Toxaphene.

10.4.3.6 If dilution is employed solely to bring a peak within the calibration range or to get the Toxaphene pattern on scale, the results for both the greater and the less concentrated extracts must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

10.4.3.7 If the DF is greater than 10, an additional extract 10 times more concentrated than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less than or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported.

10.4.3.8 When diluted, the chromatographic data for the single component pesticide must be able to be reported at greater than 10% of full scale but less than 100% of full scale.

10.4.3.9 When diluted, Toxaphene must be able to be reported at greater than 25% of full scale but less than 100% of full scale.

10.4.3.10 Samples with analytes detected at a level greater than the high calibration point must be diluted until the concentration is within the linear range established during calibration, or to a maximum of 1:100,000.

10.4.3.11 If the concentration is still above the high calibration standard concentration after the dilution of 1:100,000, the Contractor shall contact SMO immediately.

10.4.3.12 Sample dilutions must be made quantitatively. Dilute the sample extract with hexane.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Analytes

11.1.1.1 The laboratory will identify single component analyte peaks based on the RT windows established during the initial calibration sequence. Single component analytes are identified when peaks are observed in the RT window for the analyte on both GC columns.

11.1.1.2 A set of five major peaks is selected for Toxaphene. RT windows for each peak are determined from the initial calibration analysis. Identification of Toxaphene in the sample is based on pattern recognition in conjunction with the elution of five sample peaks within the RT windows of the corresponding peaks of the standard on both GC columns.

11.1.1.3 If Toxaphene is identified in a sample using a single-point calibration of a Toxaphene CS1 standard from initial calibration, then the sample must be reanalyzed with a five-point calibration and CS3 Toxaphene standard is required as the CCV.

11.1.1.4 The choice of the peaks used for Toxaphene identification and the recognition of those peaks may be complicated by the environmental alteration of Toxaphene, and by the presence of coeluting analytes, matrix interferences, or both. Because of the alteration of Toxaphene in the environment, it may give patterns in samples similar to, but not identical with, those of the standards.

11.1.2 Gas Chromatography/Mass Spectrometry Confirmation

11.1.2.1 Any pesticide listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits, for which a concentration is reported from analysis, must have the identification confirmed by GC/Mass Spectrometry (GC/MS) if the concentration is sufficient for that purpose. The following paragraphs are to be used as guidance in performing GC/MS confirmation. If the Contractor fails to perform GC/MS confirmation as appropriate, the EPA may require reanalysis of any affected samples at no additional cost to the EPA.

11.1.2.2 GC/MS confirmation may be accomplished by one of three general means:

- Examination of the semivolatile GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data]; or
- A second analysis of the semivolatile extract; or
- Analysis of the pesticide extract, following any solvent exchange and concentration steps that may be necessary.

11.1.2.3 The semivolatile GC/MS analysis procedures outlined in Exhibit D – Semivolatile Organic Compounds Analysis are based on the injection into the instrument of approximately 10 ng of a target analyte in a 2 µL volume. The semivolatile CRQL values in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 3 – Semivolatiles Target Analyte List and Contract Required Quantitation Limits are based on the sample concentration that corresponds to an on-column concentration (extract concentration) of 5 ng/µL of target analyte. Although these are quantitation limits, and the detection of analytes and

generation of reproducible mass spectra will routinely be possible at levels 3-10 times lower, the sample matrix may prevent detection of target analytes at less than 5 ng/µL. If any single component pesticide has an on-column concentration of greater than or equal to 5 ng/µL for both columns, then GC/MS confirmation is required. Similarly, for Toxaphene, if an individual peak concentration is greater than or equal to 125 ng/µL for both columns, then GC/MS confirmation is required.

11.1.2.3.1 For water samples prepared according to the method described in Section 10.1.1, 10 ng/2 µL corresponds to a sample concentration of 50 µg/L for single component pesticides, and a sample concentration of 1250 µg/L for Toxaphene.

11.1.2.3.2 For soil/sediment samples prepared according to the method described in Section 10.1.2, the corresponding sample concentration is 1,700 µg/kg for single component pesticides and 42,000 µg/kg for Toxaphene.

11.1.2.4 In order to confirm the identification of Toxaphene, the laboratory must also analyze a reference standard for Toxaphene. In order to demonstrate the ability of the GC/MS system to identify Toxaphene, the concentration of the standard should be 125 ng/µL.

11.1.2.5 To facilitate the confirmation of the single component pesticide analytes from the semivolatile library search data, the Contractor may wish to include these analytes in the semivolatile continuing calibration standard at a concentration of 5.0 ng/µL or less. Do not include Toxaphene in the semivolatile initial and continuing calibration standard. If added to this GC/MS standard, the response factors, RTs, etc., for these analytes would be reported on the GC/MS quantitation report, but not on the GC/MS calibration data reporting forms. As only a single concentration of each analyte would be analyzed, no linearity (%RSD) or %D criteria would be applied to the response factors for these additional analytes.

11.1.2.6 The Contractor is advised that library search results from the NIST (2011 release or later) mass spectral library will not likely list the name of the pesticide analyte as it appears in this analytical method; hence, the mass spectral interpretation specialist is advised to compare the Chemical Abstracts Service (CAS) Registry Numbers for the pesticides to those from the library search routine.

11.1.2.7 If the analyte cannot be confirmed from the semivolatile library search data for the original semivolatile GC/MS analysis, the Contractor may analyze another aliquot of the semivolatile sample extract after further concentration of the aliquot. This second aliquot must either be analyzed as part of a routine semivolatile GC/MS analysis, including instrument performance checks (DFTPP) and calibration standards containing the pesticides as described in Section 11.1.2.4, or it must be analyzed along with separate reference standards for the analytes to be confirmed.

11.1.2.8 If the analyte cannot be confirmed by either the procedures in Sections 11.1.2.5 or 11.1.2.7, then an aliquot of the extract prepared for the GC/ECD analysis must be analyzed by GC/MS, following any necessary solvent exchange and concentration steps. As in Section 11.1.2.4, analysis of a reference standard is required if the GC/MS continuing calibration standard does not contain the analyte to be confirmed.

11.1.2.9 Regardless of which of the three approaches above is used for GC/MS confirmation, the appropriate blank must also be analyzed by GC/MS to demonstrate that the presence of the analyte was not the result of laboratory contamination. If the confirmation is based on the analysis of the semivolatile extract, then the semivolatile method blank extracted with the sample must also be analyzed. If the confirmation is based on the analysis of the extract prepared for the GC/ECD analysis, then the pesticide method blank extracted with the sample must be analyzed.

11.1.2.10 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above, and the concentration calculated from the GC/ECD analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form 1-OR with one of the laboratory-defined qualifiers ("X", "Y", or "Z"). In this instance, define the qualifier explicitly in the SDG Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.

11.1.2.11 For GC/MS confirmation of single component analytes, the required deliverables are copies of the library search results (best TIC matches) or analyte spectrum and the spectrum of the reference standard. For Toxaphene, spectra of five characteristic peaks are required for both the sample component and the reference standard.

11.1.2.12 The purpose of the GC/MS analysis for the single component pesticides is for identification. The purpose of the GC/MS analysis for Toxaphene is to confirm the presence of chlorinated camphenes. The GC/MS analytical results for the pesticides shall not be used for quantitation and the GC/MS results shall not be reported on Form 1-OR and Form 10-OR. The exception noted in Section 11.1.2.10 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Quantitative Analysis

11.2.1 Data Processing Procedure

11.2.1.1 Target analytes identified shall be quantitated by the external standard method.

11.2.1.2 Quantitation for all analytes and surrogates must be performed and reported for each GC column.

11.2.1.3 Manual integration of peaks (e.g., measuring peak height with a ruler) is <u>only</u> permitted when accurate electronic integration of peaks cannot be done. If manual integration of peaks is required, it must be documented in the SDG Narrative.

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The

hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits, and surrogates.

11.2.1.4 The Contractor must quantitate each single component analyte and CF from the most recent initial calibration. Do not use the analyses of the Individual Standard Mixtures used to demonstrate calibration verification for quantitation of samples.

11.2.1.5 If Toxaphene is identified and the peak concentrations are calculated using a single-point calibration standard CS3 from the initial calibration, the Contractor must reanalyze the sample and quantitate with a valid five-point calibration.

NOTE: An estimated concentration (reported with an "S" flag) of the initial detection for a Toxaphene using a single-point calibration standard will be quantitated using the CF, of five major peaks, from the specific single-point calibration standard.

11.2.1.6 The chromatograms of all samples (including LCSs and MS/MSDs), standards, and required blanks must be reviewed by a qualified pesticide analyst before they are reported.

11.2.2 Target Analyte Calculations

11.2.2.1 Calculate the sample concentration and on-column concentration of the pesticides and surrogates by using the following equations:

11.2.2.2 Water

EQ. 14 Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_X}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{out}}{CV_{in}\times E}\right)_1\left(\frac{CV_{out}}{CV_{in}\times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in}\times E}\right)_n$$

WHERE,

A_x = Peak area or peak height of the compound or Toxaphene peak to be measured

$\overline{CF}$ = Mean Calibration Factor determined from the initial calibration for the peak to be measured, in area/ng

V_i = Volume of extract injected, in µL

V_t = Volume of extract produced by the preparation process (extraction and concentration), and before cleanup, in µL

V_o = Volume of the water sample extracted, in mL

NOTE: For instrument and sulfur blanks, assume a volume of 1,000 mL.

CV_{out} = Volume of extract produced by a cleanup process (cleanup and concentration), in µL

CV_{in} = Volume of extract subjected to a cleanup process, in µL

E = The efficiency of the cleanup process expressed as a fraction of the material that passes through or is not mechanically lost during the cleanup step (e.g., 50% efficiency must be expressed as 0.50)

DF = Dilution Factor, which is defined as follows:

$$DF = \frac{\mu L \text{ most concentrated extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

The $\overline{CF}$s used in Equations 14-16 are those from the most recent initial calibration. If the $\overline{CF}$s used to determine the linearity of the initial calibration were based on peak area, then the concentration of the analyte in the sample must be based on peak area. Similarly, if peak height was used to determine linearity, use peak height to determine the concentration in the sample.

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated concentration by 1000.

EQ. 15 On-Column Concentration

$$\text{On-Column Concentration (ng/}\mu\text{L)} = \frac{(A_x)}{(\overline{CF})(V_i)}$$

WHERE,

A_x, $\overline{CF}$ = As given in EQ. 14

V_i = Volume of extract injected (µL). (If a single injection is made onto two columns, use 1/2 the volume in the syringe as the volume injected onto each column.)

11.2.2.3 Soil/Sediment

EQ. 16 Soil/Sediment Concentration

$$\text{Concentration (}\mu\text{g/kg)} = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

A_x, $\overline{CF}$, V_i, V_t, CV_{out}, CV_{in}, E = As given in EQ. 14

W_t = Weight of the original soil sample extracted, in g

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

11.2.2.4 The lower of the two concentrations calculated for each single component pesticide is reported on Form 1-OR. In addition, the concentrations calculated for both the GC columns are reported on Form 10-OR, along with a %D comparing the two concentrations. The %D is calculated using the following equation:

EQ. 17 Percent Difference Between Concentrations on both GC Columns

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

WHERE,

$Conc_H$ = The higher of the two concentrations for the target analyte in question

$Conc_L$ = The lower of the two concentrations for the target analyte in question

NOTE: Using this equation will result in %D values that are always positive.

11.2.3 Toxaphene

11.2.3.1 The quantitation of Toxaphene must be accomplished by comparing the heights or the areas of each of the five major peaks of the sample with the $\overline{CF}$ for the same peaks established during the initial calibration sequence. The concentration of Toxaphene is calculated by using Equations 14 or 16, where A_x is the area for each of the major peaks. The concentration of each peak is determined and then a mean concentration for the five major peaks is determined on each column.

11.2.3.2 The reporting requirement for Toxaphene is similar to that for the single component analytes, except that the lower mean concentration (from five peaks) is reported on Form 1-OR, and the two mean concentrations reported on Form 10-OR. The two mean concentrations are compared by calculating the %D using Equation 17.

11.2.4 Contract Required Quantitation Limit Calculations

11.2.4.1 Water

EQ. 18 Water and TCLP/SPLP Leachate Sample Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL}) \left(\frac{V_x}{V_o}\right) \left(\frac{V_t}{V_y}\right) (DF) \left(\frac{CV_{out}}{CV_{in} \times E}\right)_1 \left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

V_o, V_t, DF, CV_{out}, CV_{in}, E = As given in EQ. 14

Contract CRQL = The CRQL value reported in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits

V_x = Method required sample volume (1,000 mL)

V_y = Method required concentrated extract volume (10,000 µL)

NOTE: Convert units to mg/L for TCLP leachates by dividing the final calculated CRQL by 1000.

11.2.4.2 Soil/Sediment

EQ. 19 Soil/Sediment Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL})\left(\frac{W_x}{W_t \times S}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

V_t, DF, CV_{out}, CV_{in}, E	=	As given in EQ. 16
Contract CRQL	=	As given in EQ. 18
W_x	=	Method required sample weight (30 g)
W_t	=	Weight of sample extracted, in g
S	=	% Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)
V_y	=	Method required concentrated extract volume (10,000 µL)

11.2.5 Deuterated Monitoring Compound Recoveries

Not applicable to this method.

11.2.6 Surrogate Recoveries

11.2.6.1 The concentrations for surrogate compounds are calculated by using Equations 14 or 16. Use the $\overline{CF}$s (Equation 4) from the initial calibration. If two Individual Standard Mixtures are used, $\overline{CF}$s from Individual Standard Mixture A are to be used.

11.2.6.2 The recoveries of the surrogates are calculated for each GC column according to Equation 13.

11.2.6.3 The recovery limits for the surrogates are 30-150% for both surrogate compounds.

11.2.6.4 Surrogate recovery data from both GC columns are reported (see Exhibit B – Reporting and Deliverables Requirements).

11.3 Technical Acceptance Criteria for Sample Analysis

The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

11.3.1 Samples must be analyzed under the GC/ECD operating conditions in Section 9.1. The instrument must have met all initial calibration, CCV, and blank technical acceptance criteria. Samples must be cleaned up, when required, with GPC meeting the technical acceptance criteria for GPC calibration and GPC calibration verification. Samples must be cleaned-up using Florisil that meets the technical acceptance criteria for Florisil Cartridge Performance Check. Sample analysis must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and Individual Standard Mixture(s), as described in Section 9.4.2.

11.3.2 Samples must be extracted and analyzed within the contract holding times.

11.3.3 The LCS associated with the samples must meet the LCS technical acceptance criteria.

11.3.4 The samples must have an associated method blank meeting the technical acceptance criteria for method blanks. If a sulfur cleanup blank is associated with the samples, that blank must meet the sulfur cleanup blank technical acceptance criteria.

11.3.5 The RT for each of the surrogates must be within the RT window (Section 9.3.4.4) for both GC columns.

11.3.6 The %R for the surrogates must be between 30-150%, inclusive. Up to one surrogate per sample may fail this criteria per column.

NOTE: The surrogate recovery requirements do not apply to a sample that has been diluted.

11.3.7 No target analyte concentration may exceed the upper limit concentration of the initial calibration or else extracts must be diluted and reanalyzed.

11.3.8 The identification of single component pesticides by GC methods is based primarily on RT data. The RT of the apex of a peak can only be verified from an on-scale chromatogram. The identification of Toxaphene by GC methods is based primarily on recognition of the pattern of RTs displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component analytes and Toxaphene.

11.3.8.1 When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low-point standard of the initial calibration associated with those analyses.

11.3.8.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

11.3.8.3 Chromatograms must display the largest peak of Toxaphene detected in the sample at less than full scale.

11.3.8.4 If an extract must be diluted, chromatograms must display single component pesticides between 10-100% of full scale.

11.3.8.5 If an extract must be diluted, chromatograms must display Toxaphene between 25-100% of full scale.

11.3.8.6 For any sample or blank, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

11.3.8.7 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample analysis technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or associated with a contaminated method blank or sulfur cleanup blank will require re-extraction and reanalysis at no additional cost to the EPA. Any samples analyzed that do not meet the technical acceptance criteria will require re-extraction and/or reanalysis at no additional cost to the EPA.

11.4.2 If the sample analysis technical acceptance criteria are not met, check calculations, surrogate solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria, in which case, the affected samples must be reanalyzed at no additional cost to the EPA after the corrective action.

11.4.3 The extracts from samples that were cleaned up by GPC using an automated injection system, and have both surrogate recoveries outside the lower surrogate acceptance limits, must be checked to assure that the proper amount was injected on the GPC column. If insufficient volume was injected, the sample must be reprepared and reanalyzed at no additional cost to the EPA.

11.4.4 If sample chromatograms have a high baseline or interfering peaks, inspect the system to determine the cause of the problem (e.g., carryover, column bleed, dirty ECD, contaminated gases, leaking septum, etc.). After correcting the problem, analyze an instrument blank to demonstrate that the system is functioning properly. Reanalyze the sample extracts. If the problem with the samples still exists, then those samples must be re-extracted and reanalyzed. Samples that cannot be made to meet the given specifications after one re-extraction and minimum three-step cleanup (GPC, Florisil, and sulfur cleanups) are reported in the SDG Narrative and do not require further analysis.

11.4.5 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Re-extract and reanalyze the sample. EXCEPTION: If surrogate recoveries in a sample used for an MS/MSD were outside the acceptable criteria, then it should be re-extracted/reanalyzed only if surrogate recoveries met the acceptance criteria in <u>both</u> the MS/MSD analyses.
- If the surrogate recoveries meet the acceptance criteria in the re-extracted/reanalyzed sample, then the problem was within the Contractor's control. Therefore, submit only data from the re-extraction/reanalysis.
- If the surrogate recoveries fail to meet the acceptance windows in the re-extracted/reanalyzed sample, then submit data from both analyses. Distinguish between the initial analysis and the re-extraction/reanalysis on all deliverables, using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.6 If the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume, the Contractor shall contact SMO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Summary

There are two types of blanks required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may also be required if some, but not all of the samples are subjected to sulfur cleanup. Samples that are associated with a sulfur cleanup blank are also associated with the method blank with which they were extracted. Both the method and sulfur cleanup blanks must meet the respective technical acceptance criteria for the sample analysis technical acceptance criteria to be met.

NOTE: Under no circumstances should blanks (method/instrument/sulfur cleanup) be analyzed at a dilution.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is a volume of a clean reference matrix (reagent water for aqueous samples, or purified sodium sulfate or Hydromatrix™ for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples. The leachate extraction blank shall be extracted and reported as PLEB## on Form 1A-OR.

12.1.2.2 Frequency of Method Blank

A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples [excluding MS/MSDs, Performance Evaluation (PE) samples, and LCSs]. In addition, a method blank shall:

- Be extracted by the same procedure used to extract samples; and
- Be analyzed on each GC/ECD system under the same conditions used to analyze associated samples.

12.1.2.3 Procedure for Method Blank

For water samples, measure 1.0 L volume of reagent water and spike with 1 mL of the surrogate spiking solution (Section 7.2.2.7). For soil/sediment samples, measure 30 g of sodium sulfate or Hydromatrix™ and spike with 1 mL of the surrogate spiking solution. Extract, concentrate, clean up, and analyze the method blank according to Section 10.0.

12.1.2.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.0.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.1.2.5.2 All method blanks must be prepared and analyzed at the frequency described in Section 12.1.2.2, using the procedure above and in Section 10.0 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria. Method blanks must undergo GPC cleanup, when required, on a GPC meeting the technical acceptance criteria for GPC calibration and GPC calibration verification. Method blanks must be cleaned up using Florisil meeting the technical acceptance criteria for Florisil.

12.1.2.5.3 Method blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and CS3 Standards, as described in Section 10.4.2.1.

12.1.2.5.4 The concentration of the target analytes (Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits) in the method blank must be less than the CRQL for each target analyte.

12.1.2.5.5 The method blank must meet sample technical acceptance criteria in Sections 11.3.5 and 11.3.8.

12.1.2.5.6 Surrogate recoveries must fall within the acceptance window in Table 10 – Surrogate Recovery Limits. These limits are not advisory.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.2.6.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples associated with a method blank that does not meet the method blank technical acceptance criteria will require re-extraction and reanalysis at no additional cost to the EPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.2.6.3 If surrogate recoveries in the method blank do not meet the acceptance criteria listed in Section 12.1.2.5.6, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, then the method blank and all samples associated with that method blank must be re-extracted and reanalyzed at no additional cost to the EPA.

12.1.2.6.4 If the method blank fails to meet a technical acceptance criteria other than Sections 12.1.2.5.4 and 12.1.2.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary), and reanalyze the method blank.

12.1.3 Sulfur Cleanup Blank

12.1.3.1 Summary of Sulfur Cleanup Blank

The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup and analysis procedures. The purpose of the sulfur cleanup blank is to determine the levels of contamination associated with the separate sulfur cleanup steps.

12.1.3.2 Frequency of Sulfur Cleanup Blank

The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set that required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then no separate sulfur cleanup blank is required.

12.1.3.3 Procedure for Sulfur Cleanup Blank

12.1.3.3.1 The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. The sulfur blank must also contain the surrogates at the same concentrations as the sample extracts (assuming 100.0% recovery). Therefore, add 0.60 mL of the surrogate spiking solution (Section 7.2.2.7) to 1.4 mL of hexane in a clean vial.

12.1.3.3.2 Proceed with the sulfur removal (Section 10.3.3) using the same technique (TBA sulfite or copper) as the samples associated with the blank.

12.1.3.3.3 Analyze the sulfur blank according to Section 10.4.

12.1.3.4 Calculations for Sulfur Cleanup Blank

12.1.3.4.1 Assuming that the material in the sulfur blank resulted from the extraction of a 1.0 L water sample, calculate the concentration of each analyte using Equation 14. Compare the results to the CRQL values in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

12.1.3.4.2 See Section 11.2 for the equations for the other calculations.

12.1.3.5 Technical Acceptance Criteria for Sulfur Cleanup Blank

12.1.3.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each column.

12.1.3.5.2 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Section 12.1.3.2 using the procedure in Section 12.1.3.3 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria.

12.1.3.5.3 Sulfur cleanup blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and Individual Standard Mixtures, as described in Section 10.4.2.1.

12.1.3.5.4 The concentration of the target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits) in the sulfur cleanup blank must be less than the CRQL for each target analyte.

12.1.3.5.5 The sulfur cleanup blank must meet all sample technical acceptance criteria in Sections 11.3.5 and 11.3.8.

12.1.3.5.6 Surrogate recoveries must fall within the acceptance windows in Table 10 – Surrogate Recovery Limits. These limits are not advisory.

12.1.3.6 Corrective Action for Sulfur Cleanup Blank

12.1.3.6.1 If a sulfur cleanup blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.3.6.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. Further, all samples processed with a sulfur cleanup blank that does not meet the sulfur cleanup blank technical acceptance criteria (i.e., contaminated) will require re-extraction and reanalysis at no additional cost to the EPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.3.6.3 If surrogate recoveries in the sulfur cleanup blank do not meet the technical acceptance criteria in Section 12.1.3.5.6, first reanalyze the sulfur cleanup blank. If the surrogate recoveries do not meet the technical acceptance criteria after reanalysis, then the sulfur cleanup blank and all samples associated with that sulfur cleanup blank must be reprepared/re-extracted and reanalyzed at no additional cost to the EPA.

12.1.3.6.4 If the sulfur cleanup blank fails to meet a technical acceptance criterion other than what is listed in Sections 12.1.3.5.4 and 12.1.3.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary), and reanalyze the sulfur cleanup blank.

12.1.4 Instrument Blank

12.1.4.1 Summary of Instrument Blank

An instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis, particularly with regard to carryover of analytes from standards or highly contaminated samples into other analyses.

12.1.4.2 Frequency of Instrument Blank

The first analysis in a 12-hour analysis sequence (Section 9.4) must be an instrument blank. All groups of acceptable sample analyses are to be preceded and followed by acceptable instrument blanks (Section 10.4.2.1). If more than 12 hours have elapsed

since the injection of the instrument blank that bracketed a previous 12-hour period, an instrument blank must be analyzed to initiate a new 12-hour sequence (Section 9.4.2).

12.1.4.3 Procedure for Instrument Blank

12.1.4.3.1 Prepare the instrument blank by spiking the surrogates into hexane or iso-octane for a concentration of 20.0 ng/mL of tetrachloro-m-xylene and 40.0 ng/mL of decachlorobiphenyl.

12.1.4.3.2 Analyze the instrument blank according to Section 10.4, at the frequency listed in Section 12.1.4.2.

12.1.4.4 Calculations for Instrument Blank

12.1.4.4.1 Assuming that the material in the instrument blank resulted from the extraction of a 1.0 L water sample, calculate the concentration of each analyte using Equation 14. Compare the results to the CRQL values for water samples in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

12.1.4.4.2 See Section 11.2 for the equations for the other calculations.

12.1.4.5 Technical Acceptance Criteria for Instrument Blanks

12.1.4.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed and reported independently on Form 1-OR for each GC column.

12.1.4.5.2 All instrument blanks must be prepared and analyzed at the frequency described in Section 12.1.4.2, using the procedure in Section 10.4 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria.

12.1.4.5.3 The concentration of each target analyte (Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits) in the instrument blank must be less than the CRQL for that analyte.

12.1.4.5.4 The instrument blank must meet all sample technical acceptance criteria in Sections 11.3.5 and 11.3.8.

12.1.4.5.5 Instrument blanks must be analyzed undiluted.

12.1.4.6 Corrective Action for Instrument Blank

If target analytes are detected at concentrations greater than the CRQL, or the surrogate RTs are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples that were analyzed between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be analyzed before additional data are collected. All samples (including LCSs, MS/MSDs, and PE samples) and required blanks that were analyzed after the last acceptable instrument blank must be reinjected during a valid analytical sequence and must be reported at no additional cost to the EPA.

12.2 Matrix Spike and Matrix Spike Duplicate

12.2.1 Summary of Matrix Spike and Matrix Spike Duplicate

To evaluate the effects of the sample matrix on the methods used for pesticide analyses, the EPA has prescribed a mixture of pesticide target analytes to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method.

12.2.2 Frequency of Matrix Spike and Matrix Spike Duplicate

12.2.2.1 An MS/MSD must be extracted and analyzed for every 20 field samples of a similar matrix in an SDG. MS/MSD samples must be analyzed unless otherwise specified on the Traffic Report/Chain of Custody (TR/COC) Record. If no MS/MSD samples are specified on the TR/COC Record, the Contactor shall contact SMO to confirm that MS/MSD analyses are not required.

12.2.2.2 The Contractor shall not perform MS/MSD analysis on any of the field QC or PE samples.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample volume remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify SMO that insufficient sample was received and identify the EPA sample selected for the MS/MSD analysis. SMO shall contact the EPA Region for confirmation immediately after notification. The rationale for the choice of another sample other than the one designated by the EPA shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform the requested MS/MSD, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the EPA Region for instructions. The EPA Region will either approve that no MS/MSD be performed, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the EPA Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2, the Contractor shall contact SMO. SMO will contact the EPA Region to determine which samples should have MS/MSD analysis performed on them. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.6 When a Contractor receives only PE sample(s), no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD analysis when the EPA Region did not designate a sample to be used for this purpose. SMO will notify the Contractor of the chosen sample. The Contractor must document the decision in the SDG Narrative.

12.2.3 Procedure for Preparing Matrix Spike and Matrix Spike Duplicate

12.2.3.1 For water samples, measure out two additional 1 L aliquots of the sample chosen for spiking. Fortify each with 1.0 mL of the matrix spiking solution (Section 7.2.2.8). Using a syringe or volumetric pipette, add 1 mL of surrogate standard spiking

solution to each sample (Section 7.2.2.7). Adjust the pH of the samples (if required). Extract, concentrate, cleanup, and analyze the MS/MSD according to Section 10.0.

12.2.3.2 For soil/sediment samples, weigh out two additional 30 g (to the nearest 0.1 g) aliquots of the sample chosen for spiking. Add 1.0 mL of the matrix spiking solution (Section 7.2.2.8) and 1 mL of the surrogate standard spiking solution (Section 7.2.2.7). Extract, concentrate, cleanup, and analyze the MS/MSD according to Section 10.0.

12.2.3.3 Before any MS/MSD analysis, analyze the original sample, then analyze the MS/MSD at the same concentration as the most concentrated extract for which the original sample is to be reported. For example, if the original sample is to be reported at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do not dilute MS/MSD samples further to get either spiked or non-spiked analytes within calibration range. Sample dilutions must be performed in accordance with Section 10.4.3.

12.2.4 Calculations for Matrix Spike and Matrix Spike Duplicate

12.2.4.1 Calculate the concentrations of the Matrix Spike analytes using the same equations as used for target analytes (Equations 14 and 16). Calculate the recovery of each Matrix Spike analyte using the following equation:

EQ. 20 Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spike Sample Result

SR = Original Sample Result

SA = Spike Added

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each analyte in the MS/MSD using the following equation:

EQ. 21 Relative Percent Difference

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

WHERE,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

NOTE: The vertical bars in the equation above indicate the absolute value of the difference.

12.2.5 Technical Acceptance Criteria for Matrix Spike and Matrix Spike Duplicate

12.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.2.5.2 All MS/MSDs must be prepared and analyzed at the frequency described in Section 12.2.2, using the procedure above and in Section 10.0, on a GC/ECD system meeting the initial calibration, CCV, and blank technical acceptance criteria. MS/MSDs must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, PEMs, and Individual Standard Mixture(s) (A, B, or C) as described in Section 10.4.2.1.

12.2.5.3 The MS/MSD must be extracted and analyzed within the contract required holding time.

12.2.5.4 The RT for each of the surrogates must be within the RT window as calculated in Section 9.3.4.4 for both GC columns.

12.2.5.5 The limits for spike analyte recovery and RPD are given in Table 11 – Matrix Spike Recovery and Relative Percent Difference Limits. As these limits are only advisory, no further action by the Contractor is required. However, frequent failure to meet the limits for recovery or RPD warrant investigation by the Contractor, and may result in questions from the EPA.

12.2.6 Corrective Action for Matrix Spike and Matrix Spike Duplicate

Any MS/MSD which fails to meet the technical acceptance criteria in Sections 12.2.5.1, 12.2.5.2, and 12.2.5.4 must be reanalyzed at no additional cost to the EPA.

12.3 Laboratory Control Sample

12.3.1 Summary of Laboratory Control Sample

The LCS is an internal laboratory QC sample designed to assess (on an SDG—by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit.

12.3.2 Frequency of Laboratory Control Sample

The LCS must be prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per preparation batch. The LCS must be extracted and analyzed concurrently with the samples in the SDG using the same extraction protocol, cleanup procedure, and instrumentation as the samples in the SDG.

NOTE: An LCS requires sulfur cleanup only if all samples in the specific preparation batch required this procedure.

12.3.3 Procedure for Laboratory Control Sample

12.3.3.1 For water samples, measure out 1.0 L of reagent water and spike with 1.0 mL of the LCS spiking solution (Section 7.2.2.9) and 1.0 mL of the surrogate standard spiking solution (Section 7.2.2.7). Extract, concentrate, and analyze the sample according to Section 10.0.

12.3.3.2 For soil/sediment samples, measure out 30 g of a clean reference matrix (e.g., sodium sulfate, Hydromatrix™) and spike with 1.0 mL of the LCS spiking solution (Section 7.2.2.9) and 1.0 mL of surrogate standard spiking solution (Section 7.2.2.7). Extract, concentrate, and analyze the LCS according to Section 10.0.

12.3.4 Calculations for Laboratory Control Sample

12.3.4.1 Calculate the results according to Section 11.0.

12.3.4.2 Calculate individual compound recoveries of the LCS using Equation 13.

12.3.5 Technical Acceptance Criteria for Laboratory Control Sample

12.3.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.3.5.2 The LCS must be analyzed at the frequency described in Section 12.3.2 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.3.5.3 The LCS must be prepared as described in Section 12.3.3.

12.3.5.4 The LCS must meet all sample technical acceptance criteria in Section 11.3.5.

12.3.5.5 The %R for each of the analytes in the LCS must be within the recovery limits listed in Table 12 – Laboratory Control Sample Recovery Limits.

12.3.5.6 Surrogate recoveries must fall within the acceptance windows in Table 10 – Surrogate Recovery Limits. These limits are <u>not</u> advisory.

12.3.6 Corrective Action for Laboratory Control Sample

12.3.6.1 If the LCS technical acceptance criteria for the surrogates or the LCS compound recoveries are not met, check calculations, the surrogate and LCS solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and LCS recovery criteria.

12.3.6.2 LCS technical acceptance criteria MUST be met before data are reported. LCS contamination from laboratory sources or any LCS analyzed not meeting the technical acceptance criteria will require re-extraction and reanalysis of the LCS at no additional cost to the EPA.

12.3.6.3 All samples (including MS/MSDs and PE samples) and required blanks, prepared and analyzed in an SDG with an LCS that does not meet the technical acceptance criteria, will also require re-extraction and reanalysis at no additional cost to the EPA.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed under the contract, the MDL for each single compound pesticide target analyte and Toxaphene shall be determined on each instrument used for analysis. MDL determination is matrix-specific and level-specific (i.e., the MDL shall be determined for water and soil/sediment samples). The MDLs must be determined annually thereafter or after major instrument maintenance. Major instrument maintenance includes, but is not limited to: cleaning or replacement of the detector. A new MDL study will not be required after changing the GC column, as long as the replacement has the same length, inner diameter, and stationary phase.

12.4.2 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.3 The determined concentration of the MDL must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and submitted to the EPA within seven (7) days of study completion. This schedule and the designated recipients are specified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

13.0 METHOD PERFORMANCE

Not Applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 of Exhibit D – Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 of Exhibit D – Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Acid-Base Partition Cleanup, SW-846 Method 3650B, Revision 2, December 1996.

16.2 U.S. Environmental Protection Agency, Alumina Cleanup, SW-846 Method 3610B, Revision 2, December 1996.

16.3 U.S. Environmental Protection Agency, Automated Soxhlet Extraction, SW-846 Method 3541, Revision 0, September 1994.

16.4 U.S. Environmental Protection Agency, Continuous Liquid-Liquid Extraction, SW-846 Method 3520C, Revision 3, December 1996.

16.5 U.S., Environmental Protection Agency, Gel-Permeation Cleanup, SW-846 Method 3640A, Revision 1, September 1994.

16.6 U.S. Environmental Protection Agency, Organochlorine Pesticides by Gas Chromatography, SW-846 Method 8081B Revision 2, February 2007.

16.7 U.S. Environmental Protection Agency, Pressurized Fluid Extraction (PFE), SW-846 Method 3545A, Revision 1, February 2007.

16.8 U.S. Environmental Protection Agency, Separatory Funnel Liquid-Liquid Extraction, SW-846 Method 3510C Revision 3, December 1996.

16.9 U.S. Environmental Protection Agency, Florisil Cleanup, SW-846 Method 3620C, Revision 4, July 2014.

16.10 U.S. Environmental Protection Agency, Silica Gel Cleanup, SW-846 Method 3630C, Revision 3, December 1996.

16.11 U.S. Environmental Protection Agency, Ultrasonic Extraction, SW-846 Method 3550C, Revision 3, February 2007.

16.12 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS

Systematic Name	EPA Registry Name	Synonym	CAS #
Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1.alpha.,2.alpha.,3.beta.,4.alpha.,5.beta.,6.beta.)-	.alpha.-Hexacyclo hexane	.alpha.-BHC	319-84-6
Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1.alpha.,2.beta.,3.alpha.,4.beta.,5.alpha.,6.beta.)-	.beta.-Hexacyclo hexane	.beta.-BHC	319-85-7
Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1.alpha.,2.alpha.,3.alpha.,4.beta.,5.alpha.,6.beta.)-	.delta.-Hexacyclo hexane	.delta.-BHC	319-86-8
Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1.alpha.,2.alpha.,3.beta.,4.alpha.,5.alpha.,6.beta.)-	Lindane	.gamma.-BHC (Lindane)	58-89-9
4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-	Heptachlor	Heptachlor	76-44-8
1,4:5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-, (1.alpha.,4.alpha.,4a.beta.,5.alpha.,8.alpha.,8a.beta.)-	Aldrin	Aldrin	309-00-2
2,5-Methano-2H-indeno[1,2-b]oxirene, 2,3,4,5,6,7,7-heptachloro-1a,1b,5,5a,6,6a-hexahydro-, (1aR,1bS,2R,5S,5aR,6S,6aR)-rel-	Heptachlor epoxide	Heptachlor epoxide	1024-57-3
6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide, (3.alpha.,5a.beta.,6.alpha.,9.alpha.,9a.beta.)-	.alpha.Endosulfan	Endosulfan I	959-98-8
2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aR,2R,2aS,3S,6R,6aR,7S,7aS)-rel-	Dieldrin	Dieldrin	60-57-1
Benzene, 1,1'-(dichloroethenylidene)bis[4-chloro-	p,p'-DDE	4,4'-DDE	72-55-9
2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aR,2R,2aR,3R,6S,6aS,7S,7aS)-rel, and metabolites	Endrin	Endrin	72-20-8
6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide, (3.alpha.,5a.alpha.,6.beta.,9.beta.,9a.alpha.)-	.beta.-Endosulfan	Endosulfan II	33213-65-9

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMICAL ABSTRACTS SERVICE (CAS) REGISTRY NUMBERS (CON'T)

Systematic Name	EPA Registry Name	Synonym	CAS #
Benzene, 1,1'-(2,2-dichloroethylidene)bis[4-chloro-	p,p'-DDD	4,4'-DDD	72-54-8
6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3,3-dioxide	Endosulfan sulfate	Endosulfan sulfate	1031-07-8
Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-chloro-	p,p'-DDT	4,4'-DDT	50-29-3
Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-methoxy-	Methoxychlor	Methoxychlor	72-43-5
2,5,7-Metheno-3H-cyclopenta[a]pentalen-3-one, 3b,4,5,6,6,6a-hexachlorodecahydro-, (2R,3aR,3bS,4R,5R,6aS,7S,7aR,8R)-	Endrin ketone	Endrin ketone	53494-70-5
1,2,4-Methenocyclopenta[cd]pentalene-5-carboxaldehyde, 2,2a,3,3,4,7-hexachlorodecahydro-, (1.alpha.,2.beta.,2a.beta.,4.beta.,4a.beta.,5.beta.,6a.beta.,6b.beta.,7R*)-	Endrin aldehyde	Endrinaldehyde	7421-93-4
4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-, (1R,2S,3aS,4S,7R,7aS)-rel-	Chlorodane(cis)	cis-Chlordane	5103-71-9
4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-, (1R,2R,3aS,4S,7R,7aS)-rel-	Chlorodane(trans)	trans-Chlordane	5103-74-2
Toxaphene	Toxaphene	Chlorinated camphene	8001-35-2
Benzene, 1,2,3,5-tetrachloro-4,6-dimethyl	Tetrachloro-m-xylene	2,4,5,6-Tetrachloroxylene	877-09-8
1,1'-Biphenyl, 2,2',3,3',4,4',5,5',6,6'-decachloro-	Decachlorobiphenyl	Decachloro-1,1'-biphenyl	2051-24-3

TABLE 2. CONCENTRATION LEVELS OF INITIAL CALIBRATION AND CONTINUING CALIBRATION VERIFICATION STANDARDS AND TECHNICAL ACCEPTANCE CRITERIA FOR PESTICIDES

Analyte	Concentration (ng/mL)					Maximum %RSD	Opening Maximum %D	Closing Maximum %D
	CS1	CS2	CS3	CS4	CS5			
alpha-BHC	5.0	10.	20.	40.	80.	25.0	±25.0	±25.0
gamma-BHC	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
Heptachlor	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
Endosulfan I	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
Dieldrin	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Endrin	10.	20.	40.	80.	160	20.0	±25.0	±25.0
4,4'-DDD	10.	20.	40.	80.	160	20.0	±25.0	±25.0
4,4'-DDT	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Methoxychlor	50.	100	200	400	800	20.0	±25.0	±25.0
beta-BHC	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
delta-BHC	5.0	10.	20.	40.	80.	25.0	±25.0	±25.0
Aldrin	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
Heptachlor-epoxide	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
4,4'-DDE	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Endosulfan II	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Endosulfan sulfate	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Endrin ketone	10.	20.	40.	80.	160	20.0	±25.0	±25.0
Endrin aldehyde	10.	20.	40.	80.	160	20.0	±25.0	±25.0
cis-Chlordane	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
trans-Chlordane	5.0	10.	20.	40.	80.	20.0	±25.0	±25.0
Toxaphene	500	1000	2000	4000	8000	30.0	±25.0	±25.0
Tetrachloro-m-xylene (surrogate)	5.0	10.	20.	40.	80.	20.0	±30.0	±30.0
Decachlorobiphenyl (surrogate)	10.	20.	40.	80.	160	20.0	±30.0	±30.0

NOTE: Only the exo-epoxy isomer (Isomer B) of heptachlor epoxide is used as an analytical standard.

TABLE 3. INSTRUMENT PERFORMANCE CHECK STANDARDS

	Resolution Check Mixture (RESC)	**Performance Evaluation Mixture (PEM)**
Analyte	**Concentration (ng/mL)**	
alpha-BHC	10.0	10.0
beta-BHC	10.0	10.0
delta-BHC	10.0	-
gamma-BHC	10.0	10.0
Aldrin	10.0	-
Heptachlor	10.0	-
Heptachlor-epoxide	10.0	-
cis-Chlordane	10.0	-
trans-Chlordane	10.0	-
Endosulfan I	10.0	-
Endosulfan II	20.0	-
4,4'-DDD	20.0	-
4,4'-DDE	20.0	-
4,4'-DDT	20.0	100.0
Dieldrin	20.0	-
Endrin	20.0	50.0
Endosulfan sulfate	20.0	-
Endrin ketone	20.0	-
Endrin aldehyde	20.0	-
Methoxychlor	100.0	250.0
Tetrachloro-m-xylene	10.0	20.0
Decachlorobiphenyl	20.0	20.0

TABLE 4. LOW CONCENTRATION CALIBRATION STANDARD (CS1) FOR INDIVIDUAL STANDARD MIXTURES A AND B

Individual Standard Mixture A	**Low-Point (CS1) Concentration (ng/mL)**	**Individual Standard Mixture B**	**Low-Point (CS1) Concentration (ng/mL)**
alpha-BHC	5.0	beta-BHC	5.0
gamma-BHC	5.0	delta-BHC	5.0
Heptachlor	5.0	Aldrin	5.0
Endosulfan I	5.0	Heptachlor-epoxide (exo-epoxy isomer)	5.0
Dieldrin	10.	4,4'-DDE	10.
Endrin	10.	Endosulfan II	10.
4,4'-DDD	10.	Endosulfan sulfate	10.
4,4'-DDT	10.	Endrin ketone	10.
Methoxychlor	50.	Endrin aldehyde	10.
Tetrachloro-m-xylene	5.0	cis-Chlordane	5.0
Decachlorobiphenyl	10.	trans-Chlordane	5.0
		Tetrachloro-m-xylene	5.0
		Decachloro-biphenyl	10.

TABLE 5. RETENTION TIME WINDOWS FOR
SINGLE COMPONENT ANALYTES, TOXAPHENE, AND SURROGATES

Compound	**Retention Time Window (minutes)**
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC (Lindane)	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
cis-Chlordane	± 0.07
trans-Chlordane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
4,4'-DDD	± 0.07
4,4'-DDE	± 0.07
4,4'-DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07
Endosulfan sulfate	± 0.07
Methoxychlor	± 0.07
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

TABLE 6. GAS CHROMATOGRAPH ANALYTICAL CONDITIONS

Carrier Gas:	Helium or Hydrogen 99.999% purity
Column Flow:	5 mL/min.
Make-up Gas:	Argon/Methane (P-5 or P-10) or N_2 (required)
Injector Temperature:	> 200°C
Injection Technique:	On-column
Injection Volume:	1 or 2 µl
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	0.5 min.
Temperature Ramp:	5°C to 6°C/min.
Final Temperature:	275°C
Final Hold Time:	After decachlorobiphenyl has eluted

TABLE 7. CONCENTRATION OF MATRIX SPIKE/MATRIX SPIKE DUPLICATE SPIKING, LABORATORY CONTROL SAMPLE SPIKING, AND GEL PERMEATION CHROMATOGRAPHY CALIBRATION VERIFICATION STANDARD SOLUTIONS

Analyte	**MS/MSD Spiking Solution** (µg/mL)	**LCS Spiking Solution** (µg/mL)	**GPC Calibration Verification Solution** (µg/mL)
gamma-BHC (Lindane)	0.50	0.050	0.020
Heptachlor	0.50		0.020
Aldrin	0.50		0.020
Dieldrin	1.0	0.10	0.040
Endrin	1.0	0.10	0.040
4,4'-DDT	1.0		0.040
trans-Chlordane		0.050	
Heptachlor epoxide		0.050	
4,4'-DDE		0.10	
Endosulfan sulfate		0.10	

TABLE 8. FLORISIL CARTRIDGE PERFORMANCE CHECK

COMPOUND	**QC LIMITS**
alpha-BHC	80-120
gamma-BHC (Lindane)	80-120
Heptachlor	80-120
Endosulfan I	80-120
Dieldrin	80-120
Endrin	80-120
4,4'-DDD	80-120
4,4'-DDT	80-120
Methoxychlor	80-120
TCX	80-120
DCB	80-120
2,4,5 -Trichlorophenol	<5

TABLE 9. GEL PERMEATION CHROMATOGRAPHY CALIBRATION VERIFICATION

ANALYTE	QC LIMITS
gamma-BHC (Lindane)	80-110
Heptachlor	80-110
Aldrin	80-110
Dieldrin	80-110
Endrin	80-110
4,4'-DDT	80-110

TABLE 10. SURROGATE RECOVERY LIMITS

Compound	Percent Recovery
Tetrachloro-m-xylene	30-150
Decachlorobiphenyl	30-150

TABLE 11. MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Percent Recovery Water	RPD Water	Percent Recovery Soil	RPD Soil
gamma-BHC (Lindane)	56-123	0-15	46-127	0-50
Heptachlor	40-131	0-20	35-130	0-31
Aldrin	40-120	0-22	34-132	0-43
Dieldrin	52-126	0-18	31-134	0-38
Endrin	56-121	0-21	42-139	0-45
4,4'-DDT	38-127	0-27	23-134	0-50

TABLE 12. LABORATORY CONTROL SAMPLE RECOVERY LIMITS

Analyte	Percent Recovery Water/Soil
gamma-BHC	50-120
Heptachlor epoxide	50-150
Dieldrin	30-130
4,4'-DDE	50-150
Endrin	50-120
Endosulfan sulfate	50-120
trans-Chlordane	30-130

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EXHIBIT D

AROCLORS ANALYSIS

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Exhibit D – Aroclors Analysis

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1.0 SCOPE AND APPLICATION

The analytical method that follows is designed to analyze water and soil/sediment samples from hazardous waste sites to determine the presence and concentration of the Aroclors contained in the Target Analyte List (TAL) for Aroclors in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits. The method, based on the U.S. Environmental Protection Agency (EPA) SW-846 Method 8082A, can be used for determining compound concentrations in the range from the Contract Required Quantitation Limits (CRQLs) to one million times the CRQL in these matrices, when appropriate dilutions are made. The method includes sample extraction, extract cleanup techniques, and Gas Chromatograph/Electron Capture Detector (GC/ECD) analytical methods for Aroclors.

2.0 SUMMARY OF METHOD

2.1 Water

Continuous liquid-liquid extraction or separatory funnel extraction procedures are employed for aqueous samples. A 1.0 Liter (L) aliquot of sample is spiked with the surrogate solution and extracted with methylene chloride using a separatory funnel or a continuous extractor. The methylene chloride extract is dried with anhydrous sodium sulfate (or Hydromatrix™), concentrated, and subjected to Gel Permeation Chromatography (GPC) (GPC cleanup is optional). The extract is then solvent exchanged into hexane, a 1 or 2 milliliter (mL) aliquot of the extract is subjected to a sulfuric acid cleanup, and the final volume adjusted to the same volume as the aliquot (1 mL or 2 mL). The extract is analyzed using a dual column wide-bore capillary GC/ECD.

2.2 Soil/Sediment

A 30 gram (g) aliquot of sample is spiked with the surrogates, mixed with anhydrous sodium sulfate (or Hydromatrix™), and extracted with a 1:1 (v/v) acetone/methylene chloride solvent mixture by ultrasonic extraction, Soxhlet extraction, or pressurized fluid extraction. The extract is filtered, concentrated, and subjected to GPC (GPC cleanup is optional). The extract is then solvent-exchanged into hexane, a 1 or 2 mL aliquot of the extract is subjected to a sulfuric acid cleanup, and the final volume adjusted to the same volume as the aliquot (1 mL or 2 mL). The extract is analyzed using a dual column wide-bore capillary GC/ECD.

2.3 Wipes

Not applicable to this method.

2.4 Waste

Not applicable to this method.

3.0 DEFINITIONS

See Exhibit G – Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in Gas Chromatograms. These materials must be routinely demonstrated to be free from interferences under the sample preparation and analysis conditions by analyzing instrument and method blanks. Interferences caused by phthalate esters can pose a major problem in Aroclor analysis. Because common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.2 Matrix Interferences

Matrix interferences may be caused by compounds that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. The cleanup procedures must be used to remove such interferences in order to achieve the CRQLs.

5.0 SAFETY

See Section 12.0 of Exhibit D - Introduction to Organic Analytical Methods.

5.1 Reagents

Concentrated sulfuric acid presents some hazards and is moderately toxic and extremely irritating to skin and mucous membranes. Use this reagent in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with this reagent.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternative equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 General Laboratory Equipment

6.1.1 Balances

6.1.1.1 Top loading, capable of weighing accurately to ±0.01 g.

6.1.1.2 Analytical, capable of weighing accurately to ±0.0001 g.

6.1.1.3 A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each type of balance and be accurate to ±0.01 g and ±0.0001 g, respectively. The masses that are used to check the balances daily must be checked on a monthly basis

using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '0' or Class '1') as defined by ASTM E617-97(2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified at least every five years, or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates these criteria have been met.

6.1.2 Beakers - 100 mL, 125 mL, 250 mL, and 400 mL.

6.1.3 Centrifuge, Table Top (optional).

6.1.3.1 Centrifuge Tube - 12-15 mL with 19 millimeter (mm) ground-glass joint (optional).

6.1.4 Graduated Cylinder Class A – 1.0 L and 100 mL capacity.

6.1.5 Desiccator.

6.1.6 Erlenmeyer Flasks - 250 mL.

6.1.7 Volumetric Flask, Class A – 5.0, 10, 20, 50, 100, 250, and 500 mL.

6.1.8 Magnetic Stirring Bar – Polytetrafluoroethylene (PTFE) coated, at least 4 centimeters (cm) long.

6.1.9 Ovens – drying, capable of maintaining 105°C (±5°C).

6.1.10 pH Meter - With a combination glass electrode. Calibrate according to manufacturer's instructions. The pH meter shall be calibrated prior to each use, using reference standards bracketing the range expected in samples. The pH reference standards shall be replaced when their expiration dates have passed.

6.1.11 pH Paper – Wide range.

6.1.12 Pipettes (Calibrated) - Glass volumetric, 1.0 mL or 2.0 mL. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.1.13 Spatula - Stainless steel or PTFE.

6.1.14 Syringes – 10 microliters (µL), 25 µL, 100 µL, and 1000 µL.

6.1.15 Vials and Caps - 10 mL (optional), with screw-cap and PTFE or aluminum foil liner; autosampler vial with 2 mL capacity for GC autosampler.

6.1.16 Weigh Dish – Porcelain crucibles or disposable aluminum weighing pans.

6.2 Glassware/Extraction/Cleanup Equipment

6.2.1 Automated Soxhlet Extraction System - With temperature-controlled oil bath. Silicone oil must not be used because it destroys the rubber parts. The apparatus must be used in a hood.

6.2.1.1 Cellulose or Glass Extraction Thimble, 26 mm x 60 mm.

6.2.1.2 Glass Extraction Cups.

6.2.1.3 Thimble Adapters.

6.2.1.4 Viton Seals.

6.2.2 Soxhlet Extraction, Manual

6.2.2.1 Allihn Condenser.

6.2.2.2 Soxhlet Extractor body, 40 mm ID.

6.2.2.3 Round bottom flask, 500 mL.

6.2.3 Borosilicate Glass Wool - Rinsed with methylene chloride.

6.2.4 Continuous Liquid-Liquid Extractors - Equipped with PTFE or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf extractor) or hydrophobic membrane-based extractor.

6.2.5 Drying Column – 400 mm x 19 mm ID chromatographic column with coarse frit (substitution of a small pad of disposable borosilicate glass wool for the frit will help prevent cross-contamination of sample extracts).

6.2.6 Gel Permeation Chromatography Equipment

6.2.6.1 GPC System - Systems that perform satisfactorily have been assembled from the following components: a High Performance Liquid Chromatography (HPLC) pump; an autosampler or a valving system with sample loops; and a fraction collector. All systems, whether automated or manual, must meet the calibration requirements in Section 10.3.1.3.

NOTE: Optional GPC cleanup is for all soil/sediment extracts, and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target analytes.

6.2.6.2 Chromatographic Column - 700 mm x 25 mm ID glass column. Flow is upward. To simplify switching from the ultraviolet (UV) detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

6.2.6.3 Guard Column (optional) - 5 cm, with appropriate fittings to connect to the inlet side of the analytical column.

6.2.6.4 Bio Beads (SX-3) - 200 to 400 mesh, 70 g (Bio-Rad Laboratories, Richmond, CA, or equivalent). An additional 5 g of Bio Beads is required if the optional guard column is employed. The quality of Bio Beads may vary from lot to lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they can also pass through the column screens and damage the valve.

6.2.6.5 UV Detector - Fixed wavelength [254 nanometers (nm)] with a semi-prep flow-through cell.

6.2.6.6 Strip Chart Recorder - Recording integrator or laboratory data system.

6.2.6.7 Syringe Filter Assembly, disposable – 5 micron filter discs.

NOTE: Consult your instrument operation manual to determine the proper filter disc to use in your system. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

6.2.6.8 Viscometer

6.2.7 Kuderna-Danish (K-D) Apparatus

6.2.7.1 Concentrator Tubes - 10 mL and 15 mL, graduated.

6.2.7.2 Evaporative Flasks - 500 mL.

6.2.7.3 Silicon Carbide Boiling Chips – Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride. PTFE boiling chips solvent rinsed prior to use are acceptable.

6.2.7.4 Snyder Column - Three-ball macro.

6.2.7.5 Snyder Column - Two-ball micro.

6.2.8 Nitrogen Evaporation Device - Equipped with a water bath that can be maintained at 35-40°C. To prevent the release of solvent fumes into the laboratory, the nitrogen evaporator device must be used in a hood.

6.2.9 Pressurized Fluid Extraction Device

6.2.9.1 Dionex Accelerated Solvent Extractor (ASE-300) or equivalent with appropriately-sized extraction cells. Currently, 100 mL cells are available that will accommodate greater than 30 g samples. Cells should be made of stainless steel or other material capable of withstanding the pressure requirements [2000+ pounds per square inch (psi)] necessary for this procedure.

6.2.9.2 Other system designs may be employed, provided that adequate performance can be demonstrated for the analytes and matrices of interest.

6.2.10 Separatory Funnels - 2 L with PTFE stopcock.

6.2.11 Sonabox Acoustic Enclosure (or equivalent) - For use with disruptor to decrease noise level.

6.2.12 Sulfuric Acid Cleanup

6.2.12.1 Syringe or calibrated Class A volumetric glass pipette, 1.0, 2.0, and 5.0 mL. Manufacturer’s instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.2.12.2 Vials – 1.0, 2.0, and 10 mL, glass with PTFE-lined screw-caps or crimp tops.

6.2.12.3 Vortex Mixer.

6.2.13 Ultrasonic Cell Disruptor - QSonica LLC, (53 Church Hill Road, Newtown, CT 06470) model S-4000 or equivalent ultrasonic liquid disruptor – equipped with a 3/4-inch horn and a 1/2-inch horn with a minimum output capacity of 300 watts.

NOTE 1: To ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. A rough tip surface is an indication of erosion.

NOTE 2: Follow manufacturer’s instructions for set-up.

6.2.14 Vacuum or Pressure Filtration Apparatus

6.2.14.1 Buchner Funnel.

6.2.14.2 Filter Paper – Whatman No. 42, or equivalent.

6.2.15 Water Bath - Heated, with concentric ring cover, capable of temperature control. The bath should be used in the hood.

6.3 Analytical Instrumentation

6.3.1 Gas Chromatograph

The GC system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout the temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases. The instrument must be vented to outside the facility or to a trapping system that prevents the release of contaminants into the instrument room. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants or flow controllers with rubber components are not to be used.

6.3.1.1 GCs may have difficulty in meeting certain method Quality Control (QC) requirements. This problem can be minimized by operating the injector at 200-205°C, using a borosilicate glass (not quartz) methyl silicone deactivated injector liner, and deactivating the metal parts in the injector with dichlorodimethylsilane. In some cases, using a 0.25-inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

6.3.2 Gas Chromatography Columns

Recommended Columns: Wide-bore (0.53 mm ID) fused silica GC columns may be used provided that the resolution requirements are met (Section 9.3.5); if two wide-bore (0.53 mm ID) fused silica GC columns are used, then a separate detector is required for each column. The specified analytical columns are a 30 m x 0.53 mm ID, 1.0 µm film thickness DB-1701 (J&W Scientific); SPB 1701 (Supelco); AT 1701 (Alltech); Rtx®-1701, Rtx® CLP I (Restek); CP-Sil 19CB (Chrompack); 007-1701 (Quadrex); BP-10 (SGE); or equivalent, and a 30 m x 0.53 mm ID, 0.5 to 1.0 µm film thickness DB-608 (J&W Scientific); HP-608 (Agilent); SPB-608 (Supelco); 007-608 (Quadrex); BP-608 (SGE); Rtx® CLP II; CP-Sil 8CB (Chrompack); or equivalent. A description of the GC columns used for analysis shall be provided in the SDG Narrative. Packed columns may not be used.

6.3.2.1 A capillary column is considered equivalent if:

- The column does not introduce contaminants that interfere with the identification and quantitation of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits;
- The analytical results generated using the column meet the initial calibration and continuing calibration verification (CCV) technical acceptance criteria listed in the analytical method in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits;
- The column can accept at least 16 times the low-point initial calibration concentration level in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors, without becoming overloaded; and

- The column pair chosen must have dissimilar phases and should produce different patterns to aid in Aroclor confirmation despite chromatographic interferences.

6.3.2.1.1 The column provides equal or better resolution of the analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits, than the columns listed in Section 6.3.2. Although the instructions included in the analytical method are for wide-bore capillary columns, narrower bore capillary columns may be evaluated for use. Follow manufacturer's instructions for use of its product. Document in the SDG Narrative if other columns are used by specifying the column used.

6.3.2.1.2 The Contractor must maintain documentation verifying that the alternate column met the criteria in Section 6.3.2.1. The minimum documentation is as follows:

6.3.2.1.2.1 Manufacturer provided information concerning the performance characteristics of the column.

6.3.2.1.2.2 Chromatograms and data system reports generated on the GC/ECD and used for EPA Contract Laboratory Program (CLP) analyses, including those from:

- Instrument blanks demonstrating there are no contaminants that interfere with the Aroclors analysis when using the alternate column; and
- The analysis of initial calibration and CCV standards using the alternate column.

6.3.2.1.3 Based on the Contractor-generated data described above, the Contractor shall complete a written review, signed by the Laboratory Manager, certifying that:

- The alternate column performance meets the technical acceptance criteria in Section 6.3.2;
- The low-point initial calibration standard analyses have adequate sensitivity to meet the Aroclor CRQLs;
- The high-point initial calibration standard analyses were not overloaded; and
- The alternate column does not introduce contaminants that interfere with the identification and/or quantitation of analytes listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

6.3.2.1.4 The documentation must be made available to the EPA during on-site laboratory evaluations or sent to the EPA upon request of the EPA Regional CLP Contracting Officer's Representative (COR).

6.3.2.1.5 Columns may be mounted in a press-fit Y-shaped glass 3-way union splitter or a Y-shaped fused-silica connector from a variety of commercial sources. The two columns may be mounted in an 8-inch deactivated glass injection tee. The Contractor should follow the manufacturer's recommendations for mounting 0.53 mm capillary columns in injector ports. Optionally, the dual column GC with separate autosamplers may be used for sample extractor injection.

6.3.2.1.6 The carrier gas for routine applications is helium. The Contractor may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the SDG Narrative in submissions to the EPA. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-PTFE thread sealants or flow controllers with rubber components are not to be used.

6.3.2.2 Gel Permeation Chromatography Column Preparation

Prepare the GPC column using Bio Beads. Alternate column packings may be used if: 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration verification, and 2) the column packings do not introduce contaminants/artifacts into the sample that interfere with the analysis of the Aroclor analytes. Follow the manufacturer's instructions for preparation of the GPC column.

6.3.3 Electron Capture Detector

6.3.3.1 The linearity of the response of the ECD may be greatly dependent on the flow rate of the make-up gas. The make-up gas must be P-5, P-10 (argon/methane), or nitrogen according to the instrument specification. Care must be taken to maintain stable and an appropriate flow of make-up gas to the detector. The GC/ECD system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants that may interfere with the analysis. The instrument must be vented to outside the facility or to a trapping system that prevents the release of contaminants into the instrument room.

6.3.3.2 At least annually, each ECD should be checked for radiation leakage from their Ni-63 source. Wipe tests should be conducted by wiping the inlet, outlet, and body of the ECD cell with swabs and sending the swabs for radiation tests.

6.4 Data Systems/Data Storage

A data system must be interfaced to the GC/ECD that allows the continuous acquisition and storage of data from each column throughout the duration of the chromatographic program and must permit, at a minimum, the output of time vs. intensity (peak height or peak area) data. The data system must be able to rescale chromatographic data in order to report chromatograms meeting the requirements listed within this method.

7.0 REAGENTS AND STANDARDS

The Contractor must provide all standards to be used with the contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Organic Analytical Methods, Section 11. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Reagents shall be dated with the receipt date and used on a first-in, first-out basis. The purity of the reagents shall be verified before use.

7.1 Reagents

7.1.1 Reagent Water - Reagent water is defined as water in which an interferent is not observed at or above the CRQL for each analyte of interest.

7.1.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g [1 pound (lb)] of activated carbon.

7.1.1.2 Reagent water may also be generated using a water purification system.

7.1.2 10% acetone in hexane (v/v) - Prepare by adding 10.0 mL of acetone to 90.0 mL of hexane.

7.1.3 Acetone/methylene chloride (1:1 v/v).

7.1.4 Acid Solutions: Sulfuric Acid Solution (50% v/v) - Prepare a 1:1 (v/v) solution by slowly adding 50 mL of concentrated sulfuric acid to 50 mL of reagent water.

7.1.5 Copper powder (optional) - Fine, granular. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

7.1.6 Hydromatrix™ - Diatomaceous earth-based material rinsed with methylene chloride and dried at 400°C for 4 hours in a shallow tray, cooled in a desiccator, and stored in a glass bottle.

7.1.7 Nitric Acid - Dilute, for sulfur removal with copper.

7.1.8 Sodium Hydroxide Solution (10 N) - Carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 mL.

7.1.9 Sodium sulfate - Granular anhydrous reagent grade, heated at 400°C for 4 hours, cooled in a desiccator, and stored in a glass bottle. Each lot must be extracted with hexane and analyzed by a GC/ECD to demonstrate that it is free of interference before use or must be purchased with a certification that it is free of interference.

CAUTION: AN OPEN CONTAINER OF SODIUM SULFATE MAY BECOME CONTAMINATED DURING STORAGE IN THE LABORATORY.

7.1.10 Sodium sulfite.

7.1.11 Solvents: Methylene chloride, hexane, acetone, toluene, iso-octane, petroleum ether, and methanol (optional) - Aroclor quality or equivalent. It is recommended that each lot of solvent be analyzed to demonstrate that it is free of interference before use or must be purchased with certification that it is free of interference. Methylene chloride must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid. Acidic

methylene chloride must be passed through basic alumina and then demonstrated to be free of hydrochloric acid.

7.1.12 Sulfuric acid, concentrated, 95-98% (sp. gr. 1.84).

7.1.13 Tetrabutylammonium sulfite.

7.1.14 Glycerol.

7.2 Standards

7.2.1 Stock Standard Solutions

7.2.1.1 Stock standard solutions are defined as standards that are to be used to produce working standards. They may be in the form of single compounds or mixtures. They may be prepared in methylene chloride from pure standard materials, or purchased as pre-made solutions. Prepare fresh stock standards every 6 months, or sooner if the standard has degraded or evaporated.

7.2.2 Working Standards

7.2.2.1 Aroclor Standard Mixtures

Prepare Aroclor and surrogates tetrachloro-m-xylene and decachlorobiphenyl standard solutions at a minimum of five concentration levels listed in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors. The standard solutions of each target analyte plus surrogates must be prepared individually for each Aroclor, except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture.

7.2.2.1.1 Prepare a single-point calibration for Aroclor 1221, 1232, 1242, 1248, 1254, 1262, and 1268 including surrogates at the lowest standard concentration in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors.

7.2.2.1.2 If Aroclor 1221, 1232, 1242, 1248, 1254, 1262, or 1268 are detected in a sample, then the five-point initial calibration solution for the detected Aroclor must be used for the initial calibration of the GC/ECD.

7.2.2.1.3 The Calibration Standard Mixture solutions must be prepared in either hexane or iso-octane. The solutions must be prepared every 6 months, or sooner if the solutions have degraded or concentrated.

7.2.2.1.4 The concentration of each target analyte for each calibration standard are listed in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors. These levels are based upon 10 mL final volume extracts for samples not undergoing GPC cleanup, and 5.0 mL final volume extracts for those samples undergoing GPC cleanup.

7.2.2.1.5 Other concentration levels may be used for more sensitive instrumentation and final extract volume levels. For example, in the case of Aroclor 1016, a laboratory may use a final extract volume of 10 mL for samples undergoing GPC cleanup, and a low calibration standard of 50 nanograms (ng)/mL. The alternative calibration standards and final extract volumes may be used as long as the following requirements are met:

- The Contractor can demonstrate by Method Detection Limit (MDL) studies that the MDL study calculated MDL for each target analyte is below the required CRQL for that analyte when using the laboratory's specific final volume and calibration level scheme.
- All five calibration levels are in the same ratio as that shown in Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors (e.g., if a laboratory were using a 2.5 ng/mL low standard, then the other calibration levels must be 100, 200, 400, and 800 ng/mL).

7.2.2.2 Continuing Calibration Standard

The CCV Aroclor Standards must be prepared individually, except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard solution with surrogates at or near the mid-point concentration of the initial calibration standard (Table 2 – Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors).

7.2.2.3 Gel Permeation Chromatography Calibration and Calibration Verification Solutions

7.2.2.3.1 GPC Calibration Solution

Prepare a GPC calibration solution in methylene chloride that contains the following analytes at the minimum concentrations listed below. The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

Analyte	Concentration (mg/mL)
Corn oil (CAS# 8001-30-7)	25.0
Bis(2-ethylhexyl)phthalate (CAS# 117-81-7)	0.50
Methoxychlor (CAS# 72-43-5)	0.10
Perylene (CAS# 198-55-0)	0.020
Sulfur (CAS# 7704-34-9)	0.080

NOTE: Sulfur is not very soluble in methylene chloride, but it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds.

7.2.2.3.2 GPC Calibration Verification Solution

Prepare the GPC calibration verification solution containing the Aroclors listed in Table 5 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in methylene chloride at the concentrations specified for a 5 mL GPC injection loop. See Section 10.3.1.4.3 for compound concentrations if a smaller size loop is being used. The solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.4 Surrogate Standard Spiking Solution

The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added prior to extraction to all standards, samples [including Laboratory Control Samples (LCSs)], Matrix Spike/Matrix Spike Duplicates (MS/MSDs), Performance Evaluation (PE) samples (if required), and required blanks (method/sulfur cleanup/ instrument). Prepare a surrogate standard spiking solution of 0.20 µg/mL for tetrachloro-m-xylene and 0.40 µg/mL for decachlorobiphenyl in acetone. The solution should be checked frequently for stability. The solution must be replaced every 6 months, or sooner if the solution has degraded or concentrated.

NOTE: Other concentrations for surrogate standard spiking solutions may be used, provided that the appropriate amount of each surrogate is added to all standards, samples (including LCSs), MS/MSDs, PE samples, and blanks.

7.2.2.5 Matrix Spiking Solution

Prepare a matrix spiking solution containing the Aroclors in Table 5 - Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in acetone or methanol at the concentrations specified. The solution must be replaced every 6 months, or sooner if the solution has degraded or concentrated.

7.2.2.6 Laboratory Control Sample Spiking Solution

Prepare an LCS spiking solution containing the Aroclors in Table 5 - Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions, in acetone or methanol at the concentration specified. The LCS solution must be prepared every 6 months, or sooner if the solution has degraded or concentrated.

7.2.3 Storage of Standards

7.2.3.1 Store the stock standard solutions at ≤6°C, but not frozen, in PTFE-lined, screw-cap, amber bottles/vials.

7.2.3.2 The working standards must be prepared every 6 months, or sooner if the solutions have degraded or concentrated. The working standards must be checked frequently for signs of degradation or evaporation. Store the working standard solutions at ≤6°C in PTFE-lined screw-cap, amber bottles/vials.

NOTE: Refrigeration of GPC calibration solution may cause the corn oil to precipitate. Before use, allow the solution to stand at room temperature until the corn oil dissolves. Replace this calibration solution every 6 months, or more frequently if necessary.

7.2.3.3 Standard solutions purchased from a chemical supply company as ampulated extracts in glass vials may be retained and used until the expiration date provided by the manufacturer. If no Manufacturer’s expiration date is provided, the standard solutions as ampulated extracts may be retained and used for 2 years from the preparation date. Standard solutions prepared by the Contractor that are immediately ampulated in glass vials may be retained for 2 years from the preparation date. The expiration date of the ampulated standards, upon the breaking of the glass seal, is 6 months (or sooner if the standard has degraded or evaporated).

7.2.3.4 Protect all standards from light.

7.2.3.5 Samples, sample extracts, and standards must be stored separately.

7.2.3.6 The Contractor is responsible for maintaining and verifying the integrity of standard solutions prior to use. Storage of standard solutions in the freezer may cause some compounds to precipitate. This means at a minimum, the standards must be brought to room temperature prior to use, checked for losses, and checked that all components have remained in solution. Additional steps may be necessary to ensure all components are in solution.

7.2.4 Temperature Records for Storage of Standards

7.2.4.1 The temperature of all standard storage refrigerators/freezers shall be recorded daily.

7.2.4.2 Temperature excursions shall be noted and appropriate corrective actions shall be taken to correct problems, when required.

7.2.4.3 Corrective action Standard Operating Procedures (SOPs) shall be posted on the refrigerators/freezers.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

8.1 Sample Collection and Preservation

8.1.1 Water samples

Water samples may be collected in 1.0 L (or 1.0 quart) amber glass containers, fitted with PTFE-lined screw-caps. If amber containers are not available, the samples should be protected from light.

8.1.2 Soil/Sediment Samples

Soil/sediment samples may be collected in glass containers.

8.2 Procedure for Sample and Sample Extract Storage

8.2.1 Sample Storage

The samples must be protected from light and refrigerated at ≤ 6°C, but not frozen, from the time of receipt until 60 days after delivery of a complete, reconciled data package to the EPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 Sample Extract Storage

Sample extracts must be protected from light and stored at ≤6 °C, but not frozen, until 365 days after delivery of a complete, reconciled data package to the EPA.

8.3 Contract Required Holding Times

8.3.1 Extraction of water samples by separatory funnel procedures must be completed within 5 days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction must be started within 5 days of VTSR. Extraction of soil/sediment samples shall be completed within 10 days of VTSR.

8.3.2 Analysis of sample extracts must be completed within 40 days following the start of extraction.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Initial Instrument Set-up

9.1.1 Gas Chromatograph

9.1.1.1 The GC analytical conditions are provided in Table 4 – Gas Chromatograph Analytical Conditions. Other conditions may be used, provided that all technical acceptance criteria in Sections 9.3.5, 9.4.5, and 11.3 are met.

9.1.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples (including LCSs and MS/MSDs), and required blanks (method/sulfur cleanup/instrument).

9.1.1.3 The same injection volume, 1.0 or 2.0 µL, must be used for all standards, samples (including LCSs and MS/MSDs), and required blanks (method/sulfur cleanup/instrument).

9.1.1.4 The linearity of the ECD may be greatly dependent on the flow rate of the make-up gas. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector.

9.1.1.5 Cold (ambient temperature) on-column injectors that allow injection directly onto a 0.53 mm ID column may be used as long as the initial calibration and calibration verification technical acceptance criteria are met.

9.2 Instrument Performance Check

Not applicable to this method.

9.3 Initial Calibration

9.3.1 Summary of Initial Calibration

Prior to analysis of samples (including LCSs and MS/MSDs) and required blanks (method/sulfur cleanup/instrument), each GC/ECD system must be calibrated to determine instrument sensitivity and the linearity of GC response. An initial five-point calibration is performed using Aroclors 1016 and 1260 to demonstrate the linearity of the detector response. The other seven Aroclors can be calibrated at a single low-point at a minimum, for pattern recognition. The standards for these seven Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five levels of the Aroclor 1016/1260 standards.

NOTE: All Aroclor target analytes may have five-point calibrations performed initially, prior to sample analyses. Alternately, as long as a valid five-point calibration of Aroclor 1016/1260 is present, five-point calibrations for any of the remaining Aroclor target analytes must be performed prior to reanalysis of samples known to contain the Aroclor.

9.3.2 Frequency of Initial Calibration

Each GC/ECD system must be calibrated prior to analyzing samples, after major instrument maintenance or modification is performed (e.g., column replacement or repair, cleaning or replacement of the ECD, etc.), or if the CCV technical acceptance criteria have not been met. Also, for any sample in which an Aroclor (other than Aroclor 1016 or Aroclor 1260) is detected for which a valid five-point calibration curve is not available, the sample shall be

reanalyzed following a valid five-point calibration of the specific Aroclor.

9.3.3 Procedure for Initial Calibration

9.3.3.1 Set up the GC/ECD system as described in Section 9.1. Optimize the instrumental conditions for resolution of the target analytes and sensitivity.

NOTE: Once the GC conditions have been established, the same operating conditions must be used for both calibrations and sample analyses.

9.3.3.2 All standards and instrument blanks must be allowed to warm to ambient temperature before analysis.

9.3.3.3 Prepare the initial calibration standards using the procedures, analytes, and the concentrations specified in Section 7.2.2.1.

9.3.3.4 If Aroclors other than Aroclor 1016/1260 are detected in a sample analysis, following a single-point calibration for that particular Aroclor, a separate five-point calibration must be prepared (Section 7.2.2.1) and analyzed for that particular Aroclor, followed by a reanalysis of the sample.

9.3.3.5 Analyze the initial calibration sequence which includes a five-point calibration for the Aroclor 1016/1260, and either single or five-point calibration standards for the other Aroclor analytes. All steps pertaining to the initial calibration sequence shall be performed uninterrupted with no more than the length of one chromatographic analysis separating any step. When mis-injection occurs during the initial calibration, the laboratory is allowed to perform re-injection as long as it is within the 12-hour period.

9.3.3.6 The single point calibration of Aroclors shall be at the lowest concentration (CS1) for pattern recognition at the CRQL. Each Aroclor standard shall be analyzed before the analysis of any sample. Single point Aroclor calibration may be made before or after the analysis of the five-point Aroclor calibration.

9.3.4 Calculations for Initial Calibration

9.3.4.1 During the initial calibration sequence, mean Retention Times ($\overline{RT}$s) are determined for each surrogate and five major peaks of each Aroclor (three major peaks for Aroclor 1221) on both columns.

NOTE: It is the Contractor's responsibility to ensure that DDT, DDD, and DDE do not co-elute at the same retention times as the target Aroclor analyte peaks.

9.3.4.2 For Aroclors 1016 and 1260, an RT is measured for a minimum of five peaks in each of the five calibration standards and the $\overline{RT}$ is calculated for each of the peaks as the average of the five values obtained from the five calibration standards. For Aroclor 1221, an RT is measured for three peaks for a single-point calibration standard. For Aroclors 1232, 1242, 1248, 1254, 1262, and 1268, an RT is measured for five peaks for a single-point calibration standard. For Aroclor 1262 and Aroclor 1268, the peak for DCB shall not be used as one of the five peaks for calibration. If a valid five-point calibration is present for a specific Aroclor, then an RT is measured for five peaks (three for Aroclor 1221) in each of the five calibration standards and the $\overline{RT}$ is calculated as the average of the five values for each of the peaks obtained from the five calibration standards. An RT

is measured for the surrogates in each of the five calibration standards of Aroclor 1016/1260, or from Aroclor 1016 if analyzed as a separate mixture. The surrogate $\overline{RT}$ is calculated as the average of the five values. Calculate the $\overline{RT}$ using the following equation:

EQ. 1 Mean Retention Time

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

WHERE,

RT_i = Retention Time of analyte peak

n = Total number of measurements (n=5)

9.3.4.3 An RT window is calculated for five major peaks of each Aroclor (three major peaks for Aroclor 1221) and for each surrogate using the RT window listed in Table 3 – Retention Time Windows for Analytes and Surrogates. The $\overline{RT}$s for surrogates are calculated from the five analyses of Aroclor 1016. If Aroclor 1016 and 1260 calibration standards are combined, calculate the $\overline{RT}$s for the surrogates from the combined calibration standard. Compounds are identified when peaks are observed in the RT window for the compound on both GC columns.

9.3.4.4 Five sets of Calibration Factors (CFs), one for each of the five selected peaks (three for Aroclor 1221), will be generated for the five-point initial calibration of Aroclor 1016/1260 mixture using Equation 2. Calculate the mean CFs ($\overline{CF}$s) for each set of Aroclor peaks and surrogates over the initial calibration range using Equation 3. The $\overline{CF}$s for surrogates are calculated from the five analyses of the Aroclor 1016. If Aroclor 1016 and 1260 calibration standards are combined, calculate the CFs for surrogates from the combined calibration standard.

9.3.4.5 For single-point Aroclor calibrations, calculate the CF for each selected peak.

9.3.4.6 Five sets of CFs, one for each selected peak, shall be calculated for all Aroclors that required a five-point initial calibration using Equation 2. Either peak area or peak height may be used to calculate the CFs used in the Percent Relative Standard Deviation (%RSD) equation.

9.3.4.7 Calculate the CFs, the $\overline{CF}$, and the %RSD of the CFs for each peak in a selected set of five major peaks for each Aroclor (three major peaks for Aroclor 1221) using Equations 2, 3, 4, and 5. Either peak area or peak height may be used to calculate the CFs. For example, it is permitted to calculate the CF for Aroclor 1016 based on peak area and to calculate CF for Aroclor 1260 based on peak height. It is not permitted to calculate CFs for an Aroclor from both peak area and peak height. For example, it is not permitted to calculate the CFs for the CS1 Standard for Aroclor 1260 using peak height and calculate the CS3 and CS5 Standard CFs for Aroclor 1260 using peak area.

EQ. 2 Calibration Factor

$$CF = \frac{\text{Peak area (or peak height) of the standard}}{\text{Mass Injected (ng)}}$$

9.3.4.8 Calculate the $\overline{CF}$ and the %RSD of the CF for each Aroclor analyte peak and surrogate over the initial calibration range using Equations 3 and 5.

EQ. 3 Mean Calibration Factor

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

WHERE,

CF_i = Calibration Factor

n = Total number of values (n=5)

9.3.4.9 The linearity of the instrument is determined by calculating a %RSD of the CFs from a five-point calibration curve for each of the Aroclors requiring a five-point calibration and surrogates using Equations 4 and 5.

EQ. 4 Standard Deviation of Calibration Factors

$$SD_{CF} = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{(n-1)}}$$

WHERE,

CF_i, $\overline{CF}$, n = As given in EQ. 3

EQ. 5 Percent Relative Standard Deviation of the Calibration Factors

$$\%RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

WHERE,

SD_{CF} = Standard Deviation of Calibration Factors

CF_i, $\overline{CF}$, n = As given in EQ.3

9.3.5 Technical Acceptance Criteria for Initial Calibration

All initial calibration technical acceptance criteria apply independently to each GC column.

9.3.5.1 The initial calibration sequence must be analyzed according to the procedure listed in Section 9.3.3, at the concentrations listed in Section 7.2.2.1, and at the frequency listed in Section 9.3.2. The GC/ECD operating conditions optimized in Section 9.1 must be followed.

9.3.5.2 The identification of Aroclors by GC methods is based primarily on recognition of patterns of RTs displayed on a chromatogram. Therefore, the following requirements apply to all data presented for Aroclors.

9.3.5.2.1 The chromatograms of the standards for the Aroclors analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 25% of full scale, but less than 100% of full scale.

9.3.5.2.2 If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

9.3.5.3 The %RSD of the CFs for each Aroclor peak and surrogates must be less than or equal to 20% (Table 2 - Concentration Levels of Initial Calibration and Continuing Calibration Verification Standards and Technical Acceptance Criteria for Aroclors). The %RSD requirement applies to any other Aroclor analyzed at the five-point calibration (if required in Section 9.3.3).

9.3.6 Corrective Action for Initial Calibration

9.3.6.1 If the initial calibration technical acceptance criteria are not met, reinject the initial calibration standards in sequence. If the technical acceptance criteria for the initial calibration are not met again, inspect the system for problems. It may be necessary to change the column, bake-out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

NOTE: If any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation, then the analyst should either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog.

9.3.6.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. It is recommended to refer to manufacturer's guidelines for performing detector maintenance. In the case of severe contamination, the detector may require servicing by the ECD manufacturer.

CAUTION: DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

9.3.6.3 After major maintenance is completed, the detector must be recalibrated using the initial calibration sequence.

9.3.6.4 Any samples or required blanks analyzed when the initial calibration technical acceptance criteria have not been met will require reanalysis at no additional cost to the EPA.

9.4 Continuing Calibration Verification

9.4.1 Summary of Continuing Calibration Verification

The analyses of instrument blanks and the required Aroclor CS3 standard (Section 9.4.2) constitute the calibration verification. Sample data (including LCS and MS/MSD) and required blank (method/sulfur cleanup/instrument) data are not acceptable unless bracketed by acceptable analyses of instrument blanks and the Aroclor CS3 standard (refer to Section 10.4.2.1 for the Analytical Sequence).

9.4.2 Frequency of Continuing Calibration Verification

An instrument blank and Aroclor 1016/1260 CS3 Standard Mixture must bracket one end of a 12-hour period during which sample and required blank data are collected and a second instrument blank, Aroclor 1016/1260 CS3 standard, and Aroclor CS3 standard of every other detected Aroclor(s) must bracket the other end of a 12-hour period. Each CCV must include an instrument blank and Aroclor 1016/1260 CS3 standard; additional Aroclor CS3 standards may be performed at the

laboratory's discretion. If a valid five-point calibration is available for Aroclor(s) in addition to 1016/1260, an opening CCV with an instrument blank and Aroclor 1016/1260 CS3 standard is sufficient; however, the closing CCV must include the Aroclor (CS3) matching each Aroclor detected in the sample(s) and meet opening Aroclor CCV technical acceptance criteria in Section 9.4.5.3.

9.4.2.1 Injection of an instrument blank and Aroclor 1016/1260 CS3 standard bracket the front end of the 12-hour period immediately following the initial calibration sequence (Section 9.3.3.5). The injection of any additional CS3 Aroclor standard(s) as determined by the laboratory should follow the opening instrument blank and Aroclor 1016/1260 CS3 standard. Samples (including LCSs and MS/MSDs) and required blanks (method/sulfur cleanup) may be injected for 12 hours from the injection of the instrument blank. The first injections immediately after the 12-hour period must be an instrument blank, Aroclor 1016/1260 CS3 standard (closing CCV), and CS3 standard(s) of every other detected Aroclor. A closing CCV must bracket the end of a 12-hour sequence.

9.4.2.2 The analyses of the instrument blank and CS3 Aroclor standard(s) (closing CCV) immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the technical acceptance criteria in Section 9.4.5. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and a CS3 Aroclor standard(s) (closing CCV), in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks or the required CS3 Aroclor standard(s) fails to meet the technical acceptance criteria in Section 9.4.5. The 12-hour period begins with the injection of the instrument blank.

9.4.2.3 If more than 12 hours elapse between the injections of the two instrument blanks (opening and closing CCV) that bracket a 12-hour period in which samples or required blanks are analyzed, then the time between the injection of the instrument blank (closing CCV) and the preceding sample may not exceed the length of one chromatographic analysis.

NOTE: Additional Aroclor CCV standards may be analyzed at the laboratory's discretion. The closing CCV must include Aroclor 1016/1260 CS3 and all detected Aroclors in the sample(s). When an Aroclor, other than Aroclor 1016/1260, is detected in a sample, the closing CCV CS3 standard of this detected Aroclor standard must meet opening CCV technical acceptance criteria in Section 9.4.5, if the sample was not preceded by the Aroclor included as a CS3 standard in the opening CCV. If the entire 12-hour period is not required for the analyses of all samples and blanks to be reported and all data collection is to be stopped, the sequence must end with an appropriate closing CCV combination, that is, an instrument blank, Aroclor 1016/1260 CS3 standard, and CS3 Aroclor standard(s) for every Aroclor detected in samples.

9.4.2.4 No more than 14 hours may elapse from the injection beginning the opening CCV (instrument blank) and the injection ending the closing CCV (Aroclor Standard).

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Time	Injection #	Material Injected
0 hr		Instrument Blank at end of initial calibration
		First sample if using initial calibration
		Subsequent samples
		Last Sample
12 hrs	1st injection past 12 hours	Instrument Blank
	Next injections past 12 hours	Aroclor 1016/1260 CS3 Standard
		Any other CS3 Standard (as required)
		Sample
		Subsequent samples
		Last Sample
Another 12 hrs	1st injection past 12 hours	Instrument Blank
	Next injection past 12 hours	Aroclor 1016/1260 CS3 Standard
		Any other CS3 Standard (as required)
		Sample
		Subsequent samples
		Last Sample
Another 12 hrs	1st injection past 12 hours	Instrument Blank
	Next injections past 12 hours	Aroclor 1016/1260 CS3 Standard
		Any other CS3 Standard (as required)

9.4.3 Procedure for Continuing Calibration Verification

9.4.3.1 All standards and instrument blanks must be allowed to warm to ambient temperature before analysis.

9.4.3.2 Analyze the instrument blank and the CS3 Aroclor Standard Mixture(s) according to Section 10.4 using the same injection volumes as in the initial calibration.

9.4.4 Calculations for Continuing Calibration Verification

For each analysis of the CS3 Aroclor standard(s) used to demonstrate calibration verification, calculate the %D between the CF of each Aroclor peak (and surrogate) in the standard and the corresponding $\overline{CF}$ from the Aroclor initial calibration, using the following equation:

EQ. 6 External Standard Calibration Percent Difference

$$\%D = \frac{CF - \overline{CF}}{\overline{CF}} \times 100$$

WHERE,

CF = Calibration Factor for CS3 Standard used for Calibration Verification

$\overline{CF}$ = Mean Calibration Factor

9.4.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.4.5.1 All CCV technical acceptance criteria apply independently to each column, and must meet the chromatogram criteria specified in Section 9.3.5.2.

9.4.5.2 The Aroclor 1016/1260 standards and Aroclor standards of other detected Aroclors must be analyzed at the required frequency on a GC/ECD system that has met the initial calibration technical acceptance criteria.

9.4.5.3 The RT of each of the Aroclor peaks and surrogates in the calibration verification standard must be within the RT windows determined from the initial calibration standard in Section 9.3.4.3.

9.4.5.4 For the opening CCV, the %D for each Aroclor peak and surrogates calculated from the CCV standard must not exceed ±25% and ±30.0%, respectively. For the closing CCV, the %D for each Aroclor peak and surrogates calculated from the CCV must not exceed ±50%. If the %D for the closing CCV meets the criteria for an opening CCV, then it can be used for the opening CCV of the next 12-hour period.

NOTE: When a required closing CCV of an Aroclor other than Aroclor 1016/1260 is preceded by an opening CCV of Aroclor 1016/1260 CS3 only, the %D of each Aroclor peak must not exceed ±25%. The %D requirement is waived for a closing Aroclor 1262 or Aroclor 1268 CCV standard since the DCB surrogate makes it impossible to meet the requirement.

9.4.5.5 All instrument blanks must meet the technical acceptance criteria in Section 12.1.4.5.

9.4.6 Corrective Action for Continuing Calibration Verification

9.4.6.1 If the technical acceptance criteria for the CCV are not met, inspect the system for problems and take corrective action to achieve the acceptance criteria.

9.4.6.2 Major corrective actions, such as replacing the GC column or baking out the detector, will require that a new initial calibration be performed that meets the technical acceptance criteria in Section 9.3.5.

9.4.6.3 Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the standard that originally failed the criteria and an associated instrument blank immediately after the corrective action does meet all the acceptance criteria.

9.4.6.4 If the Aroclor 1016/1260 standard does not meet technical acceptance criteria listed in Sections 9.4.5.2 and 9.4.5.3, it must be re-injected immediately. If the second injection of the Aroclor 1016/1260 standard meets the criteria, sample analysis may continue. If the second injection does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken and a new initial calibration sequence must be established before more sample data are collected.

9.4.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Section 12.1.4.5, all data collection must be stopped. Appropriate corrective action must be taken to clean out the system and an acceptable instrument blank must be analyzed before more sample data are collected.

9.4.6.6 The Contractor is reminded that analyzing an instrument blank and an Aroclor 1016/1260 standard once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to analyze instrument blanks and standards more often to avoid discarding data.

9.4.6.7 If a successful instrument blank and Aroclor 1016/1260 standard cannot be analyzed after an interruption in analysis (Section 9.4.2), an acceptable initial calibration must be analyzed before sample data may be collected. All acceptable sample analyses (including LCSs and MS/MSDs) and required blank (method/sulfur cleanup) analyses must be preceded and followed by acceptable instrument blanks and standards (opening and closing CCV) as described in Section 9.4.2.

9.4.6.8 Any samples and required blanks associated with a CCV that do not meet the technical acceptance criteria will require reanalysis at no additional cost to the EPA.

9.4.6.9 The corrective action for sample reanalysis is not required when the noncompliant analytes or surrogates, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or surrogates for which the dilution, re-extraction, or reanalysis was intended.

10.0 PROCEDURE

The Contractor must have the capability to perform all sample cleanup procedures presented in this Exhibit. The Contractor may use any of the procedures or combinations of procedures to clean up the samples prior to analysis, unless the Contractor is specifically directed by the EPA Region to use a particular cleanup procedure or combination of cleanup procedures.

The Contractor must demonstrate that each cleanup procedure is capable of producing data that meets the technical acceptance criteria for the method, including MDLs (Section 12.4) and any precision and recovery limits.

10.1 Sample Preparation

10.1.1 Water Samples

Water samples may be extracted by either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed. Allow the samples to warm to ambient temperature before extraction.

10.1.1.1 Separatory Funnel Extraction

10.1.1.1.1 For samples received in 1 L bottles, the Contractor shall mark the meniscus and transfer the entire sample into the separatory funnel. If the sample was not received in a 1 L bottle, measure out each 1 L sample aliquot in a separate graduated cylinder.

10.1.1.1.2 Measure and record the volume of sample contained in the 1 L sample bottle with water using a graduated cylinder.

10.1.1.1.3 Using a syringe or a volumetric pipette, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.2.4) to all water samples.

10.1.1.1.4 Measure and record the pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted in the SDG Narrative. Place the sample aliquot into a 2 L separatory funnel.

10.1.1.1.5 Rinse the 1 L sample bottle and/or graduated cylinder with 30 mL of methylene chloride and transfer the rinsate to the separatory funnel.

10.1.1.1.6 Add another 30 mL of methylene chloride to the separatory funnel and extract the sample by shaking the funnel for 2 minutes, with periodic venting to release excess pressure.

NOTE: The total volume of solvent used for extraction is 60 mL. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than 1/3 the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration through glass wool, centrifugation, or other physical means. Drain the methylene chloride into a 250 mL Erlenmeyer flask.

10.1.1.1.7 Add a second 60 mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Proceed to Section 10.2.

10.1.1.2 Continuous Liquid-Liquid Extraction

10.1.1.2.1 Continuous Liquid-Liquid Extraction without Hydrophobic Membrane

10.1.1.2.1.1 Follow the manufacturer's instructions for set-up.

10.1.1.2.1.2 Add 300-500 mL of methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom sidearm.

10.1.1.2.1.3 If the samples have been received in 1 L bottles, the Contractor shall mark the meniscus and transfer the entire sample into the continuous extractor. If the sample was not received in a 1 L bottle, measure out each 1.0 L sample aliquot in a separate, clean graduated cylinder and transfer the aliquot to the continuous extractor.

10.1.1.2.1.4 Using a syringe or volumetric pipette, add 1.0 mL of the surrogate standard spiking solution (Section 7.2.2.4) into the sample and mix well. Perform spiking prior to pH adjustment or any other processing steps.

10.1.1.2.1.5 Measure the pH of the sample with wide range pH paper or a pH meter and record the pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring the pH adjustment must be noted in the SDG Narrative.

NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.1.2.1.6 Rinse the graduated cylinder with a small amount of methylene chloride and transfer the rinsate to the continuous extractor. If the sample container is empty, rinse the container with 50 mL of methylene chloride and add the rinsate to the continuous extractor.

10.1.1.2.1.7 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5-15 mL/minute (recommended); optimize the extraction drip rate. Extract for a <u>minimum</u> of 18 hours.

NOTE 1: When a minimum drip rate of 10-15 mL/minute is maintained throughout the extraction, the extraction time may be reduced to a <u>minimum</u> of 12 hours. Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

NOTE 2: Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.1.2.2 Continuous Liquid-Liquid Extraction with Hydrophobic Membrane

10.1.1.2.2.1 Follow the procedure in Sections 10.1.1.2.1.1 - 10.1.1.2.1.5, but reduce the amount of methylene chloride used to 50 mL and extract for a minimum of 6 hours.

10.1.1.2.2.2 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 mL/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours.

10.1.1.2.2.3 Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion that will consume the solvent volume, preventing efficient extraction of the sample. When this occurs, add additional solvent to ensure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used. Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

10.1.1.2.2.4 Some continuous extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

10.1.1.2.2.5 If low surrogate recoveries occur, ensure that: 1) the apparatus was properly assembled to prevent leaks, 2) the drip rate/solvent cycling was optimized, and 3) there was proper cooling for condensation of solvent. Document the problem and the corrective action.

10.1.1.2.2.6 Alternate continuous extractor types that meet the requirements of the analytical method may also be used. If using alternate extractors or design types, follow the manufacturer's instructions for set-up. Optimize the extraction procedure.

10.1.2 Soil/Sediment Samples

Mix samples thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase.

10.1.2.1 Extraction of Soil/Sediment Samples

10.1.2.1.1 Three procedures are provided for the extraction of Aroclor analytes from soil/sediment samples:

- ultrasonic extraction;
- Soxhlet extraction[automated and manual]; and
- pressurized fluid extraction (PFE).

NOTE: All soil/sediment samples in a Case must be extracted by the same procedure.

10.1.2.1.2 For soil/sediment sample extractions, weigh 30-50 g of sample, to the nearest 0.1 g, into a 400 mL beaker. 30 g is ideal, as more sample may be used to compensate for high moisture content. If the system cannot accommodate 30 g of sample, a smaller sample size may be used. The specified CRQLs must be met. Adjust the amount of solvents and standards added as necessary. Document the smaller sample size in the SDG Narrative along with all steps taken to ensure sample homogeneity.

10.1.2.1.3 Add 60 g of anhydrous powdered or granulated sodium sulfate, or add 30 g of Hydromatrix™, and mix well to produce a sandy texture. Additional drying agent may be added as needed.

NOTE: For samples extracted by the PFE procedure (Section 10.1.2.1.7) the use of sodium sulfate is not recommended.

10.1.2.1.4 Add 1.0 mL of surrogate standard spiking solution to the sample, then immediately add 100 mL of 1:1 (v/v) acetone/methylene chloride. Proceed to Section 10.1.2.1.5 for ultrasonic extraction, Section 10.1.2.1.6 for automated Soxhlet extraction, or Section 10.1.2.1.7 for pressurized fluid extraction. As applicable, follow the manufacturer's instructions for use of all extraction equipment.

10.1.2.1.5 Ultrasonic Extraction

10.1.2.1.5.1 Place the bottom surface of the tip of the 3/4-inch tapered disruptor horn about 1/2 inch below the surface of the solvent, but above the sediment layer. Do not use a microtip probe.

10.1.2.1.5.2 Sonicate for 3 minutes with output at full power with pulse on (pulsing energy as opposed to continuous), and percent duty cycle knob set at 50%.

NOTE: Refer to the manufacturer's instructions for appropriate output settings.

10.1.2.1.5.3 Transfer and filter extracts through Whatman No. 42 (or equivalent) filter paper using vacuum filtration or centrifuge and decant extraction solvent.

10.1.2.1.5.4 Repeat the extraction two more times with two additional 100 mL portions of 1:1 (v/v) acetone/methylene chloride. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with a clean spatula. Transfer the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 (v/v) acetone/methylene chloride. Proceed to Section 10.2.

10.1.2.1.6 [Automated] Soxhlet Extraction

The Contractor may use either automated or non-automated Soxhlet extraction. The following procedure is based on the use of a Soxtec HT-6 automated Soxhlet extraction system. When using a different system, refer to the instructions provided by the manufacturer for the appropriate procedure.

10.1.2.1.6.1 Check the heating oil level in the automated Soxhlet unit and add oil if needed. Follow the manufacturer's instructions to set the temperature on the service unit.

10.1.2.1.6.2 Press the "MAINS" button and observe that the switch lamp is now "ON". Open the cold water tap for the reflux condensers. Adjust the flow to 2 L/minute to prevent solvent loss through the condensers.

10.1.2.1.6.3 Transfer the entire sample from the beaker (Section 10.1.2.1.4) to the thimble.

10.1.2.1.6.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

10.1.2.1.6.5 Insert the extraction cups containing boiling chips, and load each with an appropriate volume of 1:1 (v/v) acetone/methylene chloride. Using the cup holder, lower the locking handle and ensure that the safety catch engages. The cups are now clamped into position.

NOTE: The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.

10.1.2.1.6.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

10.1.2.1.6.7 Move the extraction knobs to the "RINSING" position. The thimbles will now hang above the solvent surface. Set timer for 60 minutes. Condenser valves are still open. Extract for the preset time. After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise.

10.1.2.1.6.8 When all but 2-5 mL of the solvent have been collected, open the system and remove the cups. Transfer the contents of the cups to graduated, conical-bottom glass tubes. Rinse the cups with methylene chloride and add the rinsates to the glass tubes. Proceed to Section 10.2.

10.1.2.1.7 Pressurized Fluid Extraction

10.1.2.1.7.1 Transfer the entire sample from the beaker (Section 10.1.2.1.4) to an extraction cell of the appropriate size for the aliquot.

10.1.2.1.7.2 Place the extraction cell into the instrument or autosampler tray, as described by the instrument manufacturer.

10.1.2.1.7.3 Place a pre-cleaned collection vessel in the instrument for each sample, as described by the instrument manufacturer. The total volume of the collected extract will depend on the specific instrumentation and the extraction procedure recommended by the manufacturer and may range from 0.5-1.4 times the volume of the extraction cell. Ensure that the collection vessel is sufficiently large to hold the extract.

10.1.2.1.7.4 The following are recommended extraction conditions:

Oven temperature:	100°C
Pressure:	1500-2000 psi
Static time:	5 min. (after 5 min. pre-heat equilibration)
Flush volume:	60% of the cell volume
Nitrogen purge:	60 sec. at 150 psi (purge time may be extended for larger cells)
Static cycles:	1

10.1.2.1.7.5 Optimize the extraction conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500-2000 psi should suffice. An appropriate amount of 1:1 (v/v) acetone/methylene chloride should be used to achieve the conditions in Section 10.1.2.1.7.4.

10.1.2.1.7.6 Once established, the same pressure should be used for all samples in the same SDG.

10.1.2.1.7.7 Begin the extraction according to the manufacturer's instructions. Collect each extract in a clean vial. Allow the extracts to cool after the extractions are complete. Proceed to Section 10.2.

10.2 Extract Concentration

10.2.1 Concentration by Kuderna-Danish

10.2.1.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D concentrator, if equivalency is demonstrated for all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

10.2.1.2 For water samples, transfer the extract to a K-D concentrator by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate.

10.2.1.3 For soil/sediment samples, directly transfer the extract to the K-D concentrator, if the extract is known to be dry.

10.2.1.4 Rinse the original container collecting the extract (for both water and soil/sediment samples) and the column (for water samples) with at least two 20-30 mL portions of methylene chloride to complete the quantitative transfer.

10.2.1.5 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-70°C recommended) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the

concentration in 15-30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 3-5 mL for water samples (and less than 10 mL for soil/sediment samples), remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.2.1.6 For both water and soil/sediment extracts that do not require GPC cleanup, proceed with the hexane exchange described in Section 10.2.2.

10.2.1.7 For water extracts that may require GPC cleanup, remove the Snyder column, rinse the flask and its lower joint, collect the rinsate in the concentrator tube, and adjust the volume to 10 mL with methylene chloride. Proceed to Section 10.3.1.

10.2.1.8 For soil/sediment extracts that may require GPC cleanup, it is absolutely necessary to further reduce the volume of all soil/sediment extracts to 1 mL in order to remove most of the acetone. This is best accomplished using the nitrogen evaporation technique (Section 10.2.3.2). The presence of acetone will cause a dead volume to develop in the GPC column and thus will cause a loss of surrogates and analytes during the GPC cleanups. Adjust the soil/sediment extract volume to 10 mL with methylene chloride. Proceed to Section 10.3.1.

10.2.2 Solvent Exchange into Hexane

This procedure applies to both extracts of water samples and extracts of soil/sediment samples.

10.2.2.1 With the extract in a K-D apparatus, remove the Snyder column, add 50 mL of hexane and a new boiling chip, and re-attach the Snyder column. Pre-wet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as described previously (Section 10.2.1), but increase the temperature of the water bath (80-90°C recommended) to maintain proper distillation. When the apparent volume of liquid reaches 3-5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.2.2.2 Remove the Snyder column; using 1-2 mL of hexane, rinse the flask and its lower joint into the concentrator tube. Complete quantitative transfer of the extract to a 10 mL vial by using hexane.

10.2.2.3 For samples that have <u>not</u> been subjected to GPC cleanup, adjust the volume of the hexane extract to 10 mL. For samples that <u>have</u> been subjected to GPC cleanup, concentrate the hexane extract to 5 mL using a Micro Snyder Column or nitrogen evaporation, as described in Section 10.2.3.1 or 10.2.3.2, then proceed to Section 10.3.2 for sulfuric acid cleanup.

10.2.3 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to volume before cleanup or instrumental analysis. They are the Micro Snyder Column and Nitrogen Evaporation Technique.

10.2.3.1 Micro Snyder Column Concentration

10.2.3.1.1 Add another one or two clean boiling chips to the concentrator tube and attach a two-ball Micro Snyder Column. Pre-wet the Snyder column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath

(80-90°C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and lower joint into the concentrator tube with 0.2 mL of hexane.

10.2.3.1.2 If GPC cleanup is needed and not yet performed, adjust the volume to 10 mL with methylene chloride and proceed to Section 10.3.1 for GPC cleanup. For samples that do not require GPC cleanup, adjust the volume to 10 mL with hexane and proceed to Section 10.3.2 for sulfuric acid cleanup. For samples that have already undergone GPC cleanup, adjust the volume with hexane to 5 mL and proceed to Section 10.3.2 for sulfuric acid cleanup. If no further cleanup is needed, adjust the volume with hexane to the same volume of the aliquot used for sulfuric acid and/or sulfur cleanup (1 or 2 mL) and proceed to Section 10.4 for GC/ECD analysis. Extracts may be stored at ≤6°C, but not frozen, prior to analysis.

10.2.3.2 Nitrogen Evaporation Technique

10.2.3.2.1 Place the concentrator tube in a warm water bath (30-35°C recommended) and evaporate the solvent volume to the final volume using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). DO NOT ALLOW THE EXTRACT TO GO DRY.

10.2.3.2.2 If GPC cleanup is needed and not yet performed, adjust the volume to 10 mL with methylene chloride and proceed to Section 10.3.1 for GPC cleanup. For samples that do not require GPC cleanup, adjust the volume to 10 mL with hexane and proceed to Section 10.3.2 for sulfuric acid cleanup. For samples that have already undergone GPC cleanup, adjust the volume with hexane to 5 mL and proceed to Section 10.3.2 for sulfuric acid cleanup. If no further cleanup is needed, adjust the volume with hexane to the same volume of the aliquot used for sulfuric acid and/or sulfur cleanup (1 or 2 mL) and proceed to Section 10.4 for GC/ECD analysis. Extracts may be stored at ≤6°C, but not frozen, prior to analysis.

10.2.3.2.3 Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or PTFE tubing. Plastic tubing must not be used between the carbon trap and the sample, as it may introduce interferences. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use.

10.3 Cleanup Procedures

There are three cleanup procedures specified in this method: GPC cleanup, sulfuric acid cleanup, and sulfur cleanup. Sulfur cleanup must be performed for all sample extracts contaminated with sulfur. GPC cleanup is optional for water and soil sediment extracts.

Sulfuric acid cleanup is mandatory for all extracts. Method blanks must be subjected to the same cleanup procedures as the samples (including LCSs and MS/MSDs).

10.3.1 Gel Permeation Chromatography

10.3.1.1 Introduction

GPC is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the size of the molecules to be separated.

10.3.1.2 GPC Column Preparation

Prepare the GPC column using Bio Beads. Alternate column packings may be used if: 1) the column packings have equivalent or better performance than the Bio Beads and meet the technical acceptance criteria for GPC calibration and GPC calibration verification, and 2) the column packings do not introduce contaminants/artifacts into the sample that interfere with the analysis of the Aroclor analytes. Follow the manufacturer's instructions for preparation of the GPC column.

10.3.1.3 Calibration of GPC

10.3.1.3.1 Summary of GPC Calibration

The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column.

10.3.1.3.2 Frequency of GPC Calibration

Each GPC system must be calibrated prior to processing samples under the contract, when the GPC CCV solution fails to meet criteria (Section 10.3.1.3.4), when the column is changed, when channeling occurs, and once every 7 days when in use. Also, the RT shift must be less than 5% when compared to RTs in the last calibration UV traces.

10.3.1.3.3 Procedure for GPC Calibration

Follow the manufacturer's instructions for operating the GPC system. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte RTs and must be monitored.

10.3.1.3.3.1 Using a 10 mL syringe, load the calibration solution (Section 7.2.2.3.1) onto the GPC. Determine the elution times for bis(2-ethylhexyl)phthalate, methoxychlor, and perylene. Bis(2-ethylhexyl)phthalate will elute first; perylene will elute last.

10.3.1.3.3.2 Choose a "DUMP" time that removes greater than 85% of the phthalate. Choose a "COLLECT" time so that greater than 95% of the methoxychlor is collected, and continue to collect until just prior to the elution time of sulfur. Use a "WASH" time of 10 minutes.

NOTE: The "DUMP" and "COLLECT" times must be adjusted to compensate for the difference in volume of the lines between the detector and the collection flask.

10.3.1.3.3.3 Reinject the calibration solution after appropriate "COLLECT" and "DUMP" cycles have been set, and the solvent flow and column pressure have been established.

10.3.1.3.3.4 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate problems with the system during sample processing.

10.3.1.3.3.5 Analyze a GPC blank of methylene chloride. Concentrate the methylene chloride that passed through the system during the "COLLECT" cycle using a K-D evaporator. Exchange the solvent to hexane and analyze the concentrate by GC/ECD according to the usual protocol. Assuming that the blank represents the extract from a 1 L water sample, calculate the analyte concentrations using Equation 7.

10.3.1.3.4 Technical Acceptance Criteria for GPC Calibration

10.3.1.3.4.1 The GPC system must be calibrated at the frequency described in Section 10.3.1.3.2. The UV trace must meet the following requirements:

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution;
- Corn oil and phthalate peaks should exhibit greater than 85% resolution;
- Phthalate and methoxychlor peaks should exhibit greater than 85% resolution;
- Methoxychlor and perylene peaks should exhibit greater than 85% resolution; and
- Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

10.3.1.3.4.2 The solvent flow rate and column pressure must be within the manufacturer’s specified ranges.

10.3.1.3.4.3 The RTs for bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations. Excessive RT shifts are caused by the following:

- Poor laboratory temperature control or system leaks;
- An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight; and/or
- Excessive laboratory temperatures causing outgassing of the methylene chloride.

10.3.1.3.4.4 The analyte concentrations in a GPC blank must be less than the CRQL for all target analytes in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

10.3.1.3.4.5 A copy of the two most recent UV traces of the calibration solution must be submitted with the data for the associated samples.

10.3.1.3.5 Corrective Action for GPC Calibration

10.3.1.3.5.1 If the flow rate and/or column pressure do not fall within the manufacturer’s specified ranges, a new column should be prepared.

10.3.1.3.5.2 A UV trace that does not meet the criteria in Section 10.3.1.3.4 would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

10.3.1.3.5.3 If the GPC blank exceeds the requirements in Section 10.3.1.3.4.4, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

10.3.1.4 GPC Calibration Verification

10.3.1.4.1 Summary of GPC Calibration Verification

The GPC calibration must be routinely verified with the calibration verification solution specified in Section 7.2.2.3.2.

10.3.1.4.2 Frequency of GPC Calibration Verification

10.3.1.4.2.1 The calibration verification must be performed at least once every 7 days (immediately following the GPC Calibration) whenever samples (including MS/MSDs and blanks) are cleaned up using the GPC.

10.3.1.4.2.2 Some samples may contaminate the SX-3 Bio Beads and change the retention volume of the GPC column. Therefore, system calibration and analyte recovery must be checked whenever a sample causes significant discoloration of the GPC column. Even if no darkening is visible, GPC calibration must be checked not less than once every 7 days.

10.3.1.4.3 Procedure for GPC Calibration Verification

The instructions below are for a GPC injection loop of 5 mL. If a 2 mL injection loop is used, the Contractor should adjust the volume to 4 mL instead of 10 mL before injection of the extract on the GPC.

10.3.1.4.3.1 The GPC calibration verification solution contains the Aroclor 1016 and Aroclor 1260 in methylene chloride at the concentrations in Table 5 – Concentration of Matrix Spike/Matrix Spike Duplicate Spiking, Laboratory Control Sample Spiking, and Gel Permeation Chromatography Calibration Verification Standard Solutions.

10.3.1.4.3.2 Load the 5 mL sample loop by using a 10 mL syringe containing at least 8 mL of the GPC calibration verification solution. Fractions are collected in an auto-sequence by using the GPC program established by the UV detector calibration procedure (Section 10.3.1.3).

10.3.1.4.3.3 The collected GPC calibration verification fraction is transferred to a K-D apparatus, and the collection vessel is rinsed with two additional 10 mL portions of methylene chloride to complete the transfer. The volume of methylene chloride is reduced according to Section 10.2.1. After cooling, the solvent is exchanged to hexane according to the instructions in Section 10.2.2. The final volume is adjusted to 10 mL, and the sample is analyzed by GC according to the procedure in Section 10.4. The analysis must be performed on only one of the GC columns used for sample analysis.

10.3.1.4.3.4 The recovery of each analyte must be determined for evaluation and reporting purposes. Calculate the Percent Recovery (%R) of each analyte using Equation 13.

10.3.1.4.4 Technical Acceptance Criteria for GPC Calibration Verification

The recovery of each analyte must be between 80-120%.

10.3.1.4.5 Corrective Action for GPC Calibration Verification

The Contractor may continue to use the GPC column if the technical acceptance criteria for the GPC calibration verification are met. If the recoveries are out of the acceptance criteria, the columns must be replaced and the GPC recalibrated according to the instructions in Section 10.3.1.3.3 before proceeding with any GPC cleanup on samples (including LCSs and MS/MSDs) and required method blanks.

10.3.1.5 Daily Ultraviolet Calibration Check (Optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (Section 7.2.2.3.1) and the UV detector calibration procedure (Section 10.3.1.3.3). The UV detector should be used to monitor the elution times for the phthalate, methoxychlor, and perylene, in that order. The precalibrated GPC program should "DUMP" greater than 85% of the phthalate and should "COLLECT" greater than 95% of the methoxychlor and perylene. Significant changes in elution times of the analytes (e.g., greater than 0.5 minutes) indicate that the column is out of calibration and must be recalibrated or replaced.

10.3.1.6 Sample Extract Cleanup by GPC

10.3.1.6.1 Summary of GPC Cleanup

10.3.1.6.1.1 It is very important to have consistent laboratory temperatures during an entire GPC analysis, which could be 24 hours or more. If temperatures are not consistent, RTs will shift, and the "DUMP" and "COLLECT" times determined by the calibration standard will no longer will be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C.

10.3.1.6.1.2 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of 1:1 (v/v) glycerol/water solution must be diluted and loaded into several loops. Similarly, extracts containing more than manufacturer recommended non-volatile residue must be diluted and loaded into several loops. The non-volatile residue may be determined by evaporating a 100 µL aliquot of the extract to dryness in a tared aluminum weighing pan, or another suitable container.

10.3.1.6.1.3 Systems using automated injection devices to load the sample on the column must be carefully monitored to assure that the required amount is injected onto the column. Viscous extracts or extracts containing large amounts of non-volatile residue will cause problems with injecting the proper amount of sample extract onto the column using automated injection systems. After the sample extract has been processed, the remaining sample extract in an injection vial must be checked to assure that the proper amount of extract was injected on the column before

proceeding with the extract cleanup. If the proper amount of extract was not injected, the sample must be reprepared at no additional cost to the EPA, and the sample extract must be either diluted and loaded into several loops, or the sample extract must be injected manually.

10.3.1.6.2 Frequency of Sample Extract Cleanup by GPC

GPC cleanup may be performed at least once for each soil/sediment or water extract that contains high molecular weight contaminants that interfere with the analysis of the target analytes and all associated QC samples (blanks, LCSs, and MS/MSDs) must be subjected to this procedure. GPC cleanup on the method blank must be performed after all associated samples have been cleaned up (GPC sequence: calibration, sample 1, sample 2, etc., method blank, calibration verification).

10.3.1.6.3 Procedure for Sample Extract Cleanup by GPC

10.3.1.6.3.1 Particles greater than 5 microns may scratch the valve, which may result in a system leak and cross-contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Draw the sample extract through the filter assembly and into the 10 mL syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container (e.g., a 15 mL culture tube with a PTFE-lined screw-cap).

10.3.1.6.3.2 Alternatively, draw the extract into the syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 mL of extract into a 10 mL syringe.

NOTE 1: Some GPC instrument manufacturers recommend using a smaller micron size filter disc. In this instance, follow the manufacturer’s recommended operating instructions.

NOTE 2: INTRODUCTION OF PARTICULATES OR GLASS WOOL INTO THE GPC SWITCHING VALVES MAY REQUIRE FACTORY REPAIR OF THE APPARATUS.

10.3.1.6.3.3 Follow the manufacturer’s instructions for operation of the GPC system being utilized. A 2 mL injection loop may be used in place of a 5 mL injection loop. If a 2 mL injection loop is used, concentrate the extract to 4 mL instead of 10 mL, and then inject 4 mL instead of 10 mL.

10.3.1.6.3.4 If the sample is difficult to load, part of the system may be blocked. Take appropriate corrective action, following the manufacturer’s recommendations. The problem must be resolved prior to loading sample extracts.

10.3.1.6.3.5 After loading each sample loop, wash the loading port with methylene chloride to minimize cross-contamination. Inject approximately 10 mL of methylene chloride to rinse the common tubes.

10.3.1.6.3.6 After loading the samples, process each sample using the "COLLECT" and "DUMP" cycle times established in Section 10.3.1.

10.3.1.6.3.7 Collect each sample in a 250 mL Erlenmeyer flask covered with aluminum foil to reduce solvent evaporation, or directly into a K-D evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

- Change in solvent flow rate, caused by channeling in the column or changes in column pressure;
- Increase in column operating pressure due to the accumulation of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used; and/or
- Leaks in the system or significant variances in room temperature.

10.3.1.6.3.8 After the appropriate GPC fraction has been collected for each sample, concentrate the extract as per Section 10.2.1 and proceed to solvent exchange into hexane as described in Section 10.2.2 and Sulfuric Acid cleanup in Section 10.3.2.

NOTE: Any samples that were loaded into multiple loops must be recombined before proceeding with concentration.

10.3.2 Sulfuric Acid Cleanup

10.3.2.1 Summary of Sulfuric Acid Cleanup

Sulfuric acid cleanup uses hexane solvent that will be treated with concentrated sulfuric acid. This method is used for rigorous cleanup of sample extracts prior to analysis of Aroclors. This method is used to provide accuracy in quantitation of Aroclors by eliminating elevated baselines or overly complex chromatograms.

10.3.2.2 Frequency of Sample Extract Cleanup by Sulfuric Acid

Sulfuric acid cleanup is required for all water and soil/sediment extracts.

10.3.2.3 Procedure for Sample Extract Cleanup by Sulfuric Acid

10.3.2.3.1 The volume of hexane extract used depends on the requirements of the GC autosampler used by the laboratory. If the autosampler functions reliably with 1.0 mL of sample volume, 1.0 mL of extract should be used. If the autosampler requires more than 1.0 mL of sample volume, 2.0 mL of extract should be used.

NOTE: Make sure that there is no exothermic reaction or evolution of gas prior to proceeding.

10.3.2.3.2 Using a syringe or a volumetric pipette, transfer an aliquot (1.0 or 2.0 mL) of the hexane extract to a 10 mL vial and, in a fume hood, carefully add 5.0 mL of the 1:1 (v/v) sulfuric acid/water solution.

10.3.2.3.3 Cap the vial tightly and vortex for 1 minute. A vortex must be visible in the vial.

NOTE: Stop the vortexing immediately if the vial leaks. AVOID SKIN CONTACT, AS SULFURIC ACID BURNS.

10.3.2.3.4 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored, nor should it have a visible emulsion or cloudiness.

10.3.2.3.5 If a clean phase separation is achieved, proceed to Section 10.3.2.3.8.

10.3.2.3.6 If the hexane layer is colored or the emulsion persists for several minutes, remove the sulfuric acid layer from the vial and dispose of it properly. Add another 5 mL portion of the clean 1:1 (v/v) sulfuric acid/water solution and perform another acid cleanup, beginning at Section 10.3.2.3.7.

NOTE: Do not remove any hexane from the vial at this stage of the procedure.

If the extract is no longer colored, the analyst may proceed to Section 10.3.2.3.9.

10.3.2.3.7 Vortex the sample for 1 minute and allow the phases to separate.

10.3.2.3.8 Transfer the hexane layer to a clean 10 mL vial. Take care not to include any of the acid layer in this clean vial, as it can cause damage to the analytical instrumentation.

10.3.2.3.9 Once the hexane layer is removed, perform a second "extraction" of the acid layer, as follows. Add an additional 1.0 mL of hexane to the sulfuric acid layer, cap, and vortex. This second extraction is done to ensure quantitative transfer of the Aroclors. Remove the second hexane layer and combine with the hexane from Section 10.3.2.3.8.Reduce the volume of the combined hexane layers to the original volume (1.0 mL or 2.0 mL) using an appropriate concentration technique. Analyze the extract immediately. If analysis of the extract is not performed immediately, stopper the concentrator tube and store in a refrigerator. If the extract is stored longer than 2 days, it should be transferred to a vial with a PTFE-lined screw-cap top, and labeled appropriately.

10.3.3 Sulfur Cleanup

10.3.3.1 Summary of Sulfur Cleanup

Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analyses of the later eluting Aroclors. If crystals of sulfur are evident or if the presence of sulfur is suspected, sulfur removal must be performed. Interference which is due to sulfur is not acceptable. Sulfur can be removed by one of two methods, according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract, and withdraw the solvent with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

10.3.3.2 Frequency of Sulfur Cleanup

Sulfur removal is required for all sample extracts that contain sulfur.

10.3.3.3 Procedure for Sulfur Cleanup

10.3.3.3.1 Removal of Sulfur using Tetrabutylammonium (TBA) Sulfite

The TBA sulfite procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

Add 2 mL TBA Sulfite Reagent, 1 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed, add one or two more aliquots (approximately 0.65 g) to the extract and observe. Place the samples on the wrist shaker for 45 minutes, observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5.0 mL organic free water and shake for 10-15 minutes. Place the samples into the centrifuge and spin at a setting and duration appropriate to spin down the solids. Transfer the hexane layer to a clean 10 mL vial and cap. The extract transferred to the vial still represents the 1.0 or 2.0 mL final volume.

10.3.3.3.2 Removal of Sulfur Using Copper

Add approximately 2 g of cleaned copper powder to the extract in a centrifuge or concentrator tube (2 g will fill the tube to about the 0.5 mL mark). Mix the copper and extract for at least 1 minute on a mechanical shaker. Separate the extract from the copper powder by drawing off the extract with a disposable pipette, and transfer the extract to a clean vial. The extract transferred to the vial still represents the 1.0 or 2.0 mL of extract. If upon separation of the extract, the copper appears bright, proceed to Section 10.4 and analyze the extract. If the copper changes color, repeat the sulfur removal procedure as necessary.

10.4 Gas Chromatography/Electron Capture Detector Analysis

10.4.1 Introduction

10.4.1.1 Before samples (including LCSs and MS/MSDs) and required blanks (method, sulfur cleanup, and/or instrument) can be analyzed, the instrument must meet the initial calibration and CCV technical acceptance criteria. All sample extracts, required blanks, and calibration standards must be analyzed under the same instrumental conditions. All sample extracts, required blank extracts, and standard/spiking solutions must be allowed to warm to ambient temperature before preparation/analysis. Sample analysis on two different non-equivalent GC columns (Section 6.3.2) is required for <u>all</u> samples and blanks.

10.4.1.2 Set up the GC/ECD system per the requirements in Section 9.1. Unless ambient temperature on-column injection is used, the injector must be heated to at least 200°C. The optimized GC conditions must be used.

10.4.2 Procedure for Sample Analysis by GC/ECD

The injection must be made on-column by using either automatic or manual injection. 1.0 or 2.0 µL injection volumes may be used provided that all associated standards, samples, and blanks use the same injection volume. The same injection volume must be used for

all standards, samples (including LCSs and MS/MSDs), and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2.0 µL. However, the same injection volume must be used for all analyses.

10.4.2.1 Analytical Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Time	Injection #	Material Injected
	1-12 (or 5-points of all Aroclors)	First 12 steps of the initial calibration (or 5-points of all Aroclors)
0 hr	13	Instrument Blank
	14	Aroclor 1016/1260 CS3 Standard
	15	Additional Aroclor CS3 Standard (optional)
	16	Subsequent Blank or Samples
		Last Sample
12 hrs	1st injection past 12 hours	Instrument Blank
	2nd injection past 12 hours	Aroclor 1016/1260 CS3 Standard Any other Aroclor CS3
	Subsequent injection past 12 hours	Standard (as required)
14 hrs	Last injection past 12 hours	Any other Aroclor CS3 Standard
Another 12 hrs	1st injection past 12 hours	Instrument Blank
	2nd injection past 12 hours	Aroclor 1016/1260 CS3 Standard
	Subsequent injection past 12 hours	Any other Aroclor CS3 Standard (as required)
	Injection past 12 hours	Last Sample
End of 12 hrs beginning of the next 12 hrs	2nd last injection of 12 hours	Instrument Blank
	Last injection of 12 hours	Aroclor 1016/1260 CS3 Standard and any other required Aroclor CS3

10.4.2.1.1 Injections #1 through #12 in Section 10.4.2.1 may be expanded to include all injections of initial calibration standards. The first 12 hours are counted from injection #13, not from injection #1, in the initial calibration sequence Option 1 detailed in Section 10.4.2.1. Alternately, the first 12 hours are counted from the injection of the instrument blank of an opening CCV when performed immediately after completion of the initial calibration. Samples and required blanks may be injected until 12 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that brackets the front end of the samples. If more than 12 hours elapse between the injections of two instrument blanks

that bracket a 12-hour period in which samples or required blanks are analyzed, then the time between the injection of the second instrument blank and the preceding sample may not exceed the length of one chromatographic analysis. While the 12-hour period may not be exceeded, the laboratory may analyze instrument blanks and standards more frequently, for instance, to accommodate staff working on 8-hour shifts. No more than 14 hours may elapse from the injection beginning the opening CCV (instrument blank) and the injection ending the closing CCV (Aroclor Standard).

10.4.2.1.2 After the initial calibration, the analysis sequence may continue as long as acceptable calibration verification(s) are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be analyzed at the discretion of the Contractor; however, the blanks and standards must also satisfy the criteria presented in Sections 12.0 and 9.0 in order to continue the analytical sequence.

10.4.2.1.3 An analysis sequence must also include all samples and required blank analyses, but the Contractor may decide at what point in the sequence they are to be analyzed.

10.4.2.1.4 The requirements for the analysis sequence apply to both GC columns and for all instruments used for these analyses.

10.4.3 Sample Dilutions

10.4.3.1 All samples must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography as defined in Section 11.3.

10.4.3.2 Use the results of the original analysis to determine the approximate DF required to get the largest analyte peak (for the lower of the two column concentrations) within the initial calibration range.

10.4.3.3 If more than two analyses (i.e., from the original sample extract and more than one dilution, or from the most concentrated dilution analyzed and further dilutions) are required to get all target analytes within the calibration range, contact the Sample Management Office (SMO).

10.4.3.4 If the concentration of any Aroclor peak used for quantitation is greater than the concentration of the corresponding Aroclor peak in the high standard (CS5) on both columns, then the sample must be diluted. The concentration of the target Aroclor in the diluted extract must be between the initial calibration low-point (CS1) and high-point (CS5) standards for the lower column concentration of the two analyses.

10.4.3.5 If dilution is employed solely to bring a peak within the calibration range or to get an Aroclor pattern on scale, the results for both the more and the less concentrated extracts must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

10.4.3.6 If the DF is greater than 10, an additional extract 10 times more concentrated than the diluted sample extract must be injected and reported with the sample data. If the DF is less than or equal to 10, but greater than 1, then the results of the original undiluted sample extract must also be reported.

10.4.3.7 When diluted, Aroclors must be able to be reported at greater than 25% of full scale but less than 100% of full scale.

10.4.3.8 Samples with analytes detected at a level greater than the high calibration point must be diluted until the concentration is within the linear range established during calibration, or to a maximum of 1:100,000.

10.4.3.9 If the concentration is still above the high calibration standard calibration after the dilution of 1:100,000, the Contractor shall contact SMO immediately.

10.4.3.10 Sample dilutions must be made quantitatively. Dilute the sample extract with hexane.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Analytes

11.1.1.1 The laboratory will identify analyte peaks based on the RT windows established during the initial calibration sequence.

11.1.1.2 Analytes are identified when peaks are observed in the RT window for the analyte on both GC columns.

11.1.1.3 A set of a minimum of five major peaks is selected for each Aroclor (three major peaks for Aroclor 1221). RT windows for each peak are determined from the initial calibration analysis. Identification of an Aroclor in the sample is based on pattern recognition in conjunction with the elution of a minimum of five sample peaks (three for Aroclor 1221) within the RT windows of the corresponding peaks of the standard on both GC columns.

11.1.1.4 The choice of the peaks used for Aroclor identification and the recognition of those peaks may be complicated by the environmental alteration of the Aroclors, and by the presence of coeluting analytes, matrix interferences, or both. Because of the alteration of Aroclors in the environment, Aroclors in samples may give patterns similar to, but not identical with, those of the standards.

11.1.2 Gas Chromatography/Mass Spectrometry Confirmation

11.1.2.1 Any Aroclor analyte listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits, for which a concentration is reported from a GC/ECD analysis may have the identification confirmed by GC/Mass Spectrometry (GC/MS) if the concentration is sufficient for that purpose. The following paragraphs are to be used as guidance in performing GC/MS confirmation. If the Contractor fails to perform GC/MS confirmation as appropriate, the EPA may require reanalysis of any affected samples at no additional cost to the EPA.

11.1.2.2 GC/MS confirmation may be accomplished by one of three general means:

- Examination of the semivolatile GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data]; or
- A second analysis of the semivolatile extract; or
- Analysis of the Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

11.1.2.3 If an individual peak concentration (on-column concentration) for an Aroclor is greater than or equal to 10 ng/µL for both columns, the Contractor shall contact SMO to determine whether GC/MS confirmation is required. The on-column concentration is calculated using Equation 8.

11.1.2.3.1 For water samples prepared according to the method described in Section 10.1.1, the corresponding sample concentration is 100 µg/L.

11.1.2.3.2 For soil/sediment samples prepared according to the method described in Section 10.1.2, the corresponding sample concentration is 3,300 µg/kg.

11.1.2.4 In order to confirm the identification of the target Aroclor, the Contractor must also analyze a reference standard for the analyte. In order to demonstrate the ability of the GC/MS system to identify the analyte in question, the concentration of the standard should be 50 ng/µL for Aroclors.

11.1.2.5 The Contractor is advised that library search results from the NIST (2011 release or later) mass spectral library will not likely list the name of the Aroclor analyte as it appears in this analytical method; hence, the mass spectral interpretation specialist is advised to compare the Chemical Abstracts Service (CAS) Registry numbers for the Aroclors to those from the library search routine.

11.1.2.6 If the analyte cannot be confirmed from the semivolatile library search data for the original semivolatile GC/MS analysis, the Contractor may analyze another aliquot of the semivolatile sample extract after further concentration of the aliquot. This second aliquot must either be analyzed as part of a routine semivolatile GC/MS analysis, including instrument performance checks (DFTPP) and calibration standards containing the Aroclors as described in Section 11.1.2.4, or it must be analyzed along with separate reference standards for the analyte to be confirmed.

11.1.2.7 If the analyte cannot be confirmed by the procedure in Section 11.1.2.6, then an aliquot of the extract prepared for the GC/ECD analysis must be analyzed by GC/MS, following any necessary solvent exchange and concentration steps. As in Section 11.1.2.4, analysis of a reference standard is required if the GC/MS continuing calibration standard does not contain the analyte to be confirmed.

11.1.2.8 Regardless of which of the three approaches above is used for GC/MS confirmation, the appropriate blank must also be analyzed by GC/MS to demonstrate that the presence of the analyte was not the result of laboratory contamination. If the confirmation is based on the analysis of the semivolatile extract, then the semivolatile method blank extracted with the sample must also be analyzed. If the confirmation is based on the analysis of the extract prepared for the GC/ECD analysis, then the method blank extracted with the sample must also be analyzed.

11.1.2.9 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above, and the concentration calculated from the GC/ECD analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form 1-OR with one

of the laboratory-defined qualifiers ("X", "Y", or "Z"). In this instance, define the qualifier explicitly in the SDG Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.

11.1.2.10 For GC/MS confirmation of Aroclors, spectra of three characteristic peaks are required for both the sample component and the reference standard.

11.1.2.11 The purpose of the GC/MS analysis for the Aroclors is to confirm the presence of chlorinated biphenyls in the samples. The GC/MS analytical results for the Aroclors shall not be used for quantitation and the GC/MS results shall not be reported on Form 1-OR or Form 10-OR. The exception noted in Section 11.1.2.9 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Quantitative Analysis

11.2.1 Data Processing Procedure

11.2.1.1 Target analytes identified shall be quantitated by the external standard method.

11.2.1.2 Except for an estimated value reported for an Aroclor other than 1016 or 1260, the quantitation of Aroclors must be accomplished by comparing the heights or the areas of each of five major peaks of the Aroclor (three major peaks for Aroclor 1221) in the sample with the $\overline{CF}$ for the same peaks established during the specific five-point calibration. The concentration of the target Aroclor analytes is calculated by using Equations 7 and 9, where A_x is the area for each of the major peaks of the Aroclor. The concentration of each peak is determined and then a mean concentration for five major peaks (three major peaks for Aroclor 1221) is determined on each column.

11.2.1.3 To quantitate and report the estimated concentration of an Aroclor other than 1016 or 1260, use the CF for five major peaks (three major peaks for Aroclor 1221), from the single point Aroclor calibration standard used for the Aroclor pattern recognition. It will be necessary to substitute the single CF for the $\overline{CF}$ in Equations 7, 8, and 9.

NOTE 1: The $\overline{CF}$s used for the quantitation of target Aroclors are the $\overline{CF}$s from the concentration of the specific five-point calibration.

NOTE 2: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte

List and Contract Required Quantitation Limits, and surrogates.

11.2.1.4 When an Aroclor other than 1016 or 1260 is detected in a sample, using a single point calibration, a valid five-point calibration of the specific Aroclor must be performed, followed by reanalysis of the sample or appropriately diluted sample (if the sample concentration of Aroclor exceeded calibration) with the Aroclor detected initially. If a valid five-point calibration curve is available for an Aroclor other than 1016 or 1260, the $\overline{CF}$ will be used for quantitation of the Aroclor in the sample; however, quantitation of the surrogate compounds shall use surrogate $\overline{CF}$ from the initial five-point Aroclor 1016/1260 or from Aroclor 1016 if analyzed as a separate mixture.

NOTE: An estimated concentration (reported with an "S" flag) of the initial detection for an Aroclor other than 1016 or 1260, using a single-point calibration standard will be quantitated using the CF, of five major peaks (three major peaks for Aroclor 1221), from the specific single-point calibration standard. The surrogates will be quantitated using the initial five-point Aroclor 1016/1260, or from Aroclor 1016 if analyzed as a separate mixture.

11.2.1.5 If more than one Aroclor is observed in a sample, the Contractor must choose different peaks to quantitate each Aroclor. A peak common to both analytes present in the sample must not be used to quantitate either analyte.

11.2.2 Target Analyte Calculations

11.2.2.1 Calculate the sample concentration and on-column concentration of target analytes and surrogates by using the following equations:

11.2.2.2 Water

EQ. 7 Water Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1\left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

A_x = Peak area or peak height of the peak to be measured

$\overline{CF}$ = Mean Calibration Factor determined from the initial calibration for the peak to be measured, in area/ng

V_i = Volume of extract injected, in µL

V_t = Volume of extract produced by the preparation process (extraction and concentration), and before cleanup, in µL

V_o = Volume of the water sample extracted, in mL

NOTE: For instrument and sulfur blanks, assume a volume of 1,000 mL.

CV_{out} = Volume of extract produced by a cleanup process (cleanup and concentration), in µL

CV_{in} = Volume of extract subjected to a cleanup process, in µL

E = The efficiency of the cleanup process expressed as a fraction of the material that passes through or is not mechanically lost during the cleanup step (e.g., 50% efficiency must be expressed as 0.50)

DF = Dilution Factor, which is defined as follows:

DF = $\frac{\text{µL most concentrated extract used to make dilution + µL clean solvent}}{\text{µL most concentrated extract used to make dilution}}$

If no dilution is performed, DF = 1.0.

The $\overline{CF}$s used in Equations 7-9 are those from the most recent calibration. If the $\overline{CF}$s used to determine the linearity of the initial calibration were based on peak area, then the concentration of the analyte in the sample must be based on peak area. Similarly, if peak height was used to determine linearity, use peak height to determine the concentration in the sample.

EQ. 8 On-Column Concentration

$$\text{On-Column Concentration (ng/µL)} = \frac{(A_x)}{(\overline{CF})\,(V_i)}$$

WHERE,

A_x and $\overline{CF}$ = As given in EQ. 7

V_i = Volume of extract injected (µL). (If a single injection is made onto two columns, use 1/2 the volume in the syringe as the volume injected onto each column.)

11.2.2.3 Soil/Sediment

EQ. 9 Soil/Sediment Concentration

$$\text{Concentration (µg/kg)} = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{out}}{CV_{in} \times E}\right)_1 \left(\frac{CV_{out}}{CV_{in} \times E}\right)_2 \cdots \left(\frac{CV_{out}}{CV_{in} \times E}\right)_n$$

WHERE,

A_x, $\overline{CF}$, DF, V_i, V_t, CV_{out}, CV_{in}, E = As given in EQ. 7

W_t = Weight of the soil sample extracted, in g

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

11.2.2.4 The lower <u>mean</u> concentration (from a minimum of three peaks for Aroclor 1221 and a minimum of five peaks for the remaining Aroclors) is reported on Form 1-OR, and the two mean concentrations reported on Form 10-OR. The two mean concentrations are compared by calculating the %D using the following equation:

EQ. 10 Percent Difference

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

WHERE,

$Conc_H$ = The higher of the two concentrations for the target analyte in question

$Conc_L$ = The lower of the two concentrations for the target analyte in question

NOTE: Using this equation will result in %D values that are always positive.

11.2.3 Contract Required Quantitation Limit Calculations

11.2.3.1 Water

EQ. 11 Water Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL})\left(\frac{V_x}{V_o}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{out}}{CV_{in}\times E}\right)_1\left(\frac{CV_{out}}{CV_{in}\times E}\right)_2\cdots\left(\frac{CV_{out}}{CV_{in}\times E}\right)_n$$

WHERE,

V_o, V_t, DF, CV_{out}, CV_{in}, E = As given in EQ. 7

Contract CRQL = The CRQL value reported in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits

V_x = Method required sample volume (1,000 mL)

V_y = Method required concentrated extract volume (10,000 µL)

11.2.3.2 Soil/Sediment

EQ. 12 Soil/Sediment Adjusted CRQL

$$\text{Adjusted CRQL} = (\text{Contract CRQL})\left(\frac{W_x}{W_t\times S}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{out}}{CV_{in}\times E}\right)_1\left(\frac{CV_{out}}{CV_{in}\times E}\right)_2\cdots\left(\frac{CV_{out}}{CV_{in}\times E}\right)_n$$

WHERE,

DF, V_t, CV_{out}, CV_{in}, E = As given in EQ. 7

Contract CRQL = As given in EQ. 11

W_x = Method required sample weight (30 g)

W_t = Weight of sample extracted, in g

S = % Solids/100 (Exhibit D – General Organic Analysis, Section 10.1.1)

V_y = Method required concentrated extract volume (10,000 µL)

11.2.4 Deuterated Monitoring Compound Recoveries

Not applicable to this method.

11.2.5 Surrogate Recoveries

11.2.5.1 The concentrations for surrogate compounds on each column are calculated by using Equations 7 and 9. Use the $\overline{CF}$s from a valid initial five-point calibration of Aroclor 1016/1260, or from Aroclor 1016 if analyzed as a separate mixture.

11.2.5.2 Calculate surrogate recoveries for each GC column using the following equation:

EQ. 13 Surrogate Recovery

$$\%R = \frac{(Q_d \times DF)}{Q_a}\times 100$$

WHERE,

Q_d = Quantity determined by analysis

Q_a = Quantity added

DF = Dilution Factor

11.2.5.3 The recovery limits for the surrogates are 30-150% for both surrogate compounds.

11.2.5.4 Surrogate recovery data from both GC columns are reported (see Exhibit B – Reporting and Deliverables Requirements).

11.3 Technical Acceptance Criteria for Sample Analysis

The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

11.3.1 Samples must be analyzed under the GC/ECD operating conditions in Section 9.1. The instrument must have met all initial calibration, CCV, and blank technical acceptance criteria. Sample analysis must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks and CCV standards described in Section 9.4.2.

11.3.2 Samples must be extracted and analyzed within the contract required holding times.

11.3.3 The LCS associated with the samples must meet the LCS technical acceptance criteria.

11.3.4 The samples must have an associated method blank meeting the technical acceptance criteria for method blanks. If a sulfur cleanup blank is associated with the samples, that blank must meet the sulfur cleanup blank technical acceptance criteria.

11.3.5 Surrogate compounds RT must be compared to the window established during a valid initial five-point calibration of Aroclor 1016/1260 or from Aroclor 1016 if analyzed as a separate mixture. The RT for each of the surrogates must be within the RT window (Section 9.3.4.3) for both GC columns.

11.3.6 The %R for the surrogates must be between 30-150%, inclusive. Up to one surrogate per sample may fail this criteria per column. Exception: If Aroclor 1262 or 1268 is detected in a sample, the %R of the DCB surrogate is advisory for both column analyses of the specific sample. However, %R for TCX must meet the acceptance criteria.

NOTE: The surrogate recovery requirements do not apply to a sample that has been diluted.

11.3.7 No target analyte concentration may exceed the upper limit concentration of the initial calibration or else the extract must be diluted and reanalyzed.

11.3.8 If a valid initial calibration is not available, then a five-point calibration curve specific for any identified Aroclor must be analyzed during a valid analytical sequence on the same instrument and column upon its detection in a sample.

11.3.9 The identification of Aroclors is based primarily on recognition of patterns of RTs displayed on a chromatogram. Therefore, the following requirements apply to all data presented for Aroclors.

11.3.9.1 Five peaks must be chosen for each Aroclor with the exception of Aroclor 1221, where three peaks must be chosen. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of five peaks (three for Aroclor 1221) should include at least one peak that is unique to that Aroclor.

11.3.9.2 Chromatograms must display the largest peak of any Aroclor detected in the sample at less than full scale.

11.3.9.3 If an extract must be diluted, chromatograms must display the peaks chosen for quantitation of Aroclors between 25-100% of full scale.

11.3.9.4 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample analysis technical acceptance criteria must be met before data are reported. Samples contaminated from laboratory sources or associated with a contaminated method blank or sulfur cleanup blank will require re-extraction and reanalysis at no additional cost to the EPA. Any samples analyzed that do not meet the technical acceptance criteria will require re-extraction and/or reanalysis at no additional cost to the EPA.

11.4.2 If the sample analysis technical acceptance criteria are not met, check calculations, surrogate solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria, in which case, the affected samples must be reanalyzed at no additional cost to the EPA after the corrective action. Reanalyses of the MS and MSD samples are not required for any target Aroclor qualified with an "S" flag, if this same Aroclor target is detected and reported with a five-point calibration in the original sample.

11.4.3 The extracts from samples that were cleaned up by GPC using an automated injection system, and have both surrogate recoveries outside the lower surrogate acceptance limits, must be checked to assure that the proper amount was injected on the GPC column. If insufficient volume was injected, the sample must be reprepared and reanalyzed at no additional cost to the EPA.

11.4.4 If sample chromatograms have a high baseline or interfering peaks, inspect the system to determine the cause of the problem (e.g., carryover, column bleed, dirty ECD, contaminated gases, leaking septum, etc.). After correcting the problem, analyze an instrument blank to demonstrate that the system is functioning properly. Reanalyze the sample extracts. If the problem with the samples still exists, then those samples must be re-extracted and reanalyzed. Samples that cannot be made to meet the given specifications after one re-extraction and cleanup procedures (sulfuric acid and GPC cleanups) are reported in the SDG Narrative and do not require further analysis.

11.4.5 If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was a matrix effect, take the following corrective action steps:

- Re-extract and reanalyze the sample. EXCEPTION: If surrogate recoveries in a sample used for an MS/MSD were outside the acceptance criteria, then it should be re-extracted/reanalyzed only if surrogate recoveries met the acceptance criteria in both the MS/MSD analyses.

- If the surrogate recoveries meet the acceptance windows in the re-extracted/reanalyzed sample, then the problem was within the Contractor's control. Therefore, submit only data from the re-extraction/reanalysis.
- If the surrogate recoveries fail to meet the acceptance windows in the re-extracted/reanalyzed sample, then submit data from both analyses. Distinguish between the initial analysis and the re-extraction/reanalysis on all deliverables using the suffixes in Exhibit B – Reporting and Deliverables Requirements, Table 5 – Codes for Labeling Data.

11.4.6 If the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume, the Contractor shall contact SMO.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Summary

There are two types of blanks required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may also be required if some, but not all of the samples are subjected to sulfur cleanup. Samples that are associated with a sulfur cleanup blank are also associated with the method blank with which they were extracted. Both the method and sulfur cleanup blanks must meet the respective technical acceptance criteria for the sample analysis technical acceptance criteria to be met.

NOTE: Under no circumstances should blanks (method/instrument/sulfur cleanup) be analyzed at a dilution.

12.1.2 Method Blank

12.1.2.1 Summary of Method Blank

A method blank is a volume of a clean reference matrix (reagent water for aqueous samples, or purified sodium sulfate or Hydromatrix™ for soil/sediment samples) carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.2.2 Frequency of Method Blank

A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples (excluding MS/MSDs, PE samples, and LCSs). In addition, a method blank shall:

- Be extracted by the same procedure used to extract samples; and
- Be analyzed on each GC/ECD system under the same conditions used to analyze associated samples.

12.1.2.3 Procedure for Method Blank

For water samples, measure a 1.0 L volume of reagent water and spike with 1.0 mL of the surrogate spiking solution (Section 7.2.2.4). For soil/sediment samples, measure 30 g of sodium sulfate or Hydromatrix™ and spike with 1.0 mL of the surrogate

spiking solution. Extract, concentrate, clean up, and analyze the method blank according to Section 10.0.

12.1.2.4 Calculations for Method Blank

Perform data analysis and calculations according to Section 11.0.

12.1.2.5 Technical Acceptance Criteria for Method Blank

12.1.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.1.2.5.2 All method blanks must be prepared and analyzed at the frequency described in Section 12.1.2.2, using the procedure above and in Section 10.0 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria. Method blanks must undergo GPC cleanup, when required, on a GPC meeting the technical acceptance criteria for GPC calibration and GPC calibration verification.

12.1.2.5.3 Method blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, and required Aroclor standards, as described in Section 10.4.2.1.

12.1.2.5.4 The concentration of the target analytes, (Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits) in the method blank must be less than the CRQL for each target analyte.

12.1.2.5.5 The method blank must meet all sample technical acceptance criteria in Section 11.3.5.

12.1.2.5.6 Surrogate recoveries must fall within the acceptance window in Table 6 – Surrogate Recovery Limits. These limits are not advisory.

12.1.2.6 Corrective Action for Method Blank

12.1.2.6.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.2.6.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples associated with a method blank that does not meet the method blank technical acceptance criteria will require re-extraction and reanalysis at no additional cost to the EPA. It is the Contractor’s responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.2.6.3 If surrogate recoveries in the method blank do not meet the acceptance criteria listed in Section 12.1.2.5.6, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, then the method blank and all samples associated with that method blank must be re-extracted and reanalyzed at no additional cost to the EPA.

12.1.2.6.4 If the method blank fails to meet a technical acceptance criterion other than what is listed in Sections 12.1.2.5.4 and 12.1.2.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary), and reanalyze the method blank.

12.1.3 Sulfur Cleanup Blank

12.1.3.1 Summary of Sulfur Cleanup Blank

The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup and analysis procedures. The purpose of the sulfur cleanup is to determine the levels of contamination associated with the separate sulfur cleanup steps.

12.1.3.2 Frequency of Sulfur Cleanup Blank

The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set that required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then no separate sulfur cleanup blank is required.

12.1.3.3 Procedure for Sulfur Cleanup Blank

12.1.3.3.1 The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. The sulfur blank must also contain the surrogates at the same concentrations as the sample extracts (assuming 100.0% recovery).

12.1.3.3.2 Proceed with the sulfur removal (Section 10.3.3) using the same technique (TBA sulfite or copper) as the samples associated with the blank.

12.1.3.3.3 Analyze the sulfur blank according to Section 10.4.

12.1.3.4 Calculations for Sulfur Cleanup Blank

12.1.3.4.1 Assuming that the material in the sulfur blank resulted from the extraction of a 1.0 L water sample, calculate the concentration of each analyte using Equation 7. Compare the results to the CRQL values in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

12.1.3.4.2 See Section 11.2 for the equations for the other calculations.

12.1.3.5 Technical Acceptance Criteria for Sulfur Cleanup Blank

12.1.3.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each column.

12.1.3.5.2 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Section 12.1.3.2 using the procedure in Section 12.1.3.3 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria.

12.1.3.5.3 Sulfur cleanup blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks and required Aroclor Standards, as described in Section 10.4.2.1.

12.1.3.5.4 The concentration of the target analytes (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits) in the sulfur cleanup blank must be less than the CRQL for each target analyte.

12.1.3.5.5 The sulfur cleanup blank must meet all sample technical acceptance criteria in Section 11.3.5.

12.1.3.5.6 Surrogate recoveries must fall within the acceptance criteria in Table 6 – Surrogate Recovery Limits. These limits are not advisory.

12.1.3.6 Corrective Action for Sulfur Cleanup Blank

12.1.3.6.1 If a sulfur cleanup blank does not meet the technical acceptance criteria, the Contractor must consider the analytical system to be out of control.

12.1.3.6.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. Further, all samples processed with a sulfur cleanup blank that does not meet the sulfur cleanup blank technical acceptance criteria (i.e., contaminated) will require re-extraction and reanalysis at no additional cost to the EPA. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated.

12.1.3.6.3 If surrogate recoveries in the sulfur cleanup blank do not meet the technical acceptance criteria in Section 12.1.3.5.6, first reanalyze the sulfur cleanup blank. If the surrogate recoveries do not meet the technical acceptance criteria after reanalysis, then the sulfur cleanup blank and all samples associated with that sulfur cleanup blank must be re-prepared/ re-extracted and reanalyzed at no additional cost to the EPA.

12.1.3.6.4 If the sulfur cleanup blank fails to meet a technical acceptance criterion other than what is listed in Sections 12.1.3.5.4 and 12.1.3.5.6, then the problem is an instrument problem. Correct the instrument problem, recalibrate the instrument (if necessary), and reanalyze the sulfur cleanup blank.

12.1.4 Instrument Blank

12.1.4.1 Summary of Instrument Blank

An instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis, particularly with regard to carryover of analytes from standards or highly contaminated samples into other analysis.

12.1.4.2 Frequency of Instrument Blank

The first analysis in a 12-hour analysis sequence (Section 9.4) must be an instrument blank. All groups of acceptable sample analyses are to be preceded and followed by acceptable instrument blanks (Section 10.4.2.1). If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a

previous 12-hour period, an instrument blank must be analyzed to initiate a new 12-hour sequence (Section 9.4.2).

12.1.4.3 Procedure for Instrument Blank

12.1.4.3.1 Prepare the instrument blank by spiking the surrogates into hexane or iso-octane for a concentration of 20.0 ng/mL of tetrachloro-m-xylene and 40.0 ng/mL of decachlorobiphenyl.

12.1.4.3.2 Analyze the instrument blank according to Section 10.4, at the frequency listed in Section 12.1.4.2.

12.1.4.4 Calculations for Instrument Blank

12.1.4.4.1 Assuming that the material in the instrument blank resulted from the extraction of a 1 L water sample, calculate the concentration of each analyte using Equation 7. Compare the results to the CRQL values for water samples in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

12.1.4.4.2 See Section 11.2 for the equations for the other calculations.

12.1.4.5 Technical Acceptance Criteria for Instrument Blanks

12.1.4.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed and reported independently on Form 1-OR for each GC column.

12.1.4.5.2 All instrument blanks must be prepared and analyzed at the frequency described in Section 12.1.4.2, using the procedure in Section 10.4 on a GC/ECD system meeting the initial calibration and CCV technical acceptance criteria.

12.1.4.5.3 The concentration of each target analyte (Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits) in the instrument blank must be less than the CRQL for that analyte.

12.1.4.5.4 The instrument blank must meet all sample technical acceptance criteria in Section 11.3.5.

12.1.4.5.5 Instrument blanks must be analyzed undiluted.

12.1.4.6 Corrective Action for Instrument Blank

If target analytes are detected at concentrations greater than the CRQL, or the surrogate RTs are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples that were analyzed between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be analyzed before additional data are collected. All samples (including LCSs, MS/MSDs, and PE samples) and required blanks that were analyzed after the last acceptable instrument blank must be re-injected during a valid analytical sequence and must be reported at no additional cost to the EPA.

12.2 Matrix Spike and Matrix Spike Duplicate

12.2.1 Summary of Matrix Spike and Matrix Spike Duplicate

To evaluate the effects of the sample matrix on the methods used for Aroclor analyses, the EPA has prescribed a multi-component mixture of Aroclor 1016 and Aroclor 1260 to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method.

12.2.2 Frequency of Matrix Spike and Matrix Spike Duplicate Analysis

12.2.2.1 An MS/MSD must be extracted and analyzed for every 20 field samples of a similar matrix in an SDG. MS/MSD samples must be analyzed unless otherwise specified on the Traffic Report/Chain of Custody (TR/COC) Record. If no MS/MSD samples are specified on the TR/COC Record, the Contractor shall contact SMO to confirm that MS/MSD analyses are not required.

12.2.2.2 The Contractor shall not perform MS/MSD analysis on any of the field QC or PE samples.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample volume remaining to perform an MS/MSD, then the Contractor shall choose another sample to perform an MS/MSD analysis. At the time the selection is made, the Contractor shall notify SMO that insufficient sample was received and identify the EPA sample selected for the MS/MSD analysis. SMO shall contact the EPA Region for confirmation immediately after notification. The rationale for the choice of another sample other than the one designated by the EPA shall be documented in the SDG Narrative.

12.2.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform the requested MS/MSD, the Contractor shall immediately contact SMO to inform them of the problem. SMO will contact the EPA Region for instructions. The EPA Region will either approve that no MS/MSD be performed, or require that a reduced sample aliquot be used for the MS/MSD analysis. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.5 If it appears that the EPA Region has requested MS/MSD analysis at a greater frequency than specified in Section 12.2.2, the Contractor shall contact SMO. SMO will contact the EPA Region to determine which samples should have an MS/MSD analysis performed on them. SMO will notify the Contractor of the EPA Region’s decision. The Contractor shall document the decision in the SDG Narrative.

12.2.2.6 When a Contractor receives only PE sample(s), no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG, a sample other than the PE sample must be chosen for the MS/MSD analysis when the EPA Region did not designate samples to be used for this purpose. If the PE sample is received as an ampulated standard extract, the ampulated PE sample is not considered to be another matrix type. SMO will notify the Contractor of the chosen sample. The Contractor must document the decision in the SDG Narrative.

12.2.3 Procedure for Preparing Matrix Spike and Matrix Spike Duplicate

12.2.3.1 For water samples, measure out two additional 1.0 L aliquots of the sample chosen for spiking. Fortify each with 1.0 mL of matrix spiking solution (Section 7.2.2.5). Using a syringe or volumetric pipette, add 1.0 mL of surrogate standard spiking solution to each sample (Section 7.2.2.4). Adjust the pH of the samples (if required). Extract, concentrate, cleanup, and analyze the MS/MSD according to Section 10.0.

12.2.3.2 For soil/sediment samples, weigh out two additional 30 g (to the nearest 0.1 g) aliquots of the sample chosen for spiking. Add 1 mL of the matrix spiking solution (Section 7.2.2.5) and 1 mL of the surrogate standard spiking solution (Section 7.2.2.4). Extract, concentrate, cleanup, and analyze the MS/MSDs according to Section 10.0.

12.2.3.3 Before any MS/MSD analysis, analyze the original sample, then analyze the MS/MSD at the same concentration as the most concentrated extract for which the original sample results will be reported. For example, if the original sample is to be reported at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do <u>not</u> dilute MS/MSD samples further to get either spiked or non-spiked analytes within calibration range. Sample dilutions must be performed in accordance with Section 10.4.3.

12.2.4 Calculations for Matrix Spike and Matrix Spike Duplicate

12.2.4.1 Calculate the concentrations of the Matrix Spike analytes using the same equation as used for target analytes (Equations 7 and 9). Calculate the recovery of each Matrix Spike analyte using the following equation:

EQ. 14 Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spike Sample Result

SR = Original Sample Result

SA = Spike Added

12.2.4.2 Calculate the Relative Percent Difference (RPD) of the recoveries of each analyte in the MS/MSD using the following equation:

EQ. 15 Relative Percent Difference

$$RPD = \frac{\left|MSR - MSDR\right|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

WHERE,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

NOTE: The vertical bars in the equation above indicate the absolute value of the difference.

12.2.5 Technical Acceptance Criteria for Matrix Spike and Matrix Spike Duplicate

12.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.2.5.2 All MS/MSDs must be prepared and analyzed at the frequency described in Section 12.2.2 using the procedure above, and in Section 10.0, on a GC/ECD system meeting the initial calibration, CCV, and blank technical acceptance criteria. MS/MSDs must be bracketed at 12-hour intervals (or less) by acceptable calibration verification described in Section 10.4.2.1.

12.2.5.3 The MS/MSD must be extracted and analyzed within the contract required holding time.

12.2.5.4 The RT for each of the surrogates must be within the RT window as calculated in Section 9.3.4.3 for both GC columns.

12.2.5.5 The limits for MS analyte recovery and RPD are given in Table 7 – Matrix Spike Recovery and Relative Percent Difference Limits. As these limits are only advisory, no further action by the Contractor is required. However, frequent failure to meet the limits for recovery or RPD warrants investigation by the Contractor, and may result in questions from the EPA.

12.2.6 Corrective Action for Matrix Spike and Matrix Spike Duplicate

Any MS/MSD that fails to meet the technical acceptance criteria in Sections 12.2.5.1, 12.2.5.2, and 12.2.5.4 must be reanalyzed at no additional cost to the EPA.

12.3 Laboratory Control Sample

12.3.1 Summary of Laboratory Control Sample

The LCS is an internal laboratory QC sample designed to assess (on an SDG-by-SDG basis) the capability of the Contractor to perform the analytical method listed in this Exhibit.

12.3.2 Frequency of Laboratory Control Sample

The LCS must be prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per preparation batch. The LCS must be extracted and analyzed concurrently with the samples in the SDG using the same extraction protocol, cleanup procedures, and instrumentation as the samples in the SDG.

NOTE: An LCS requires sulfur cleanup only if all samples in the specific preparation batch required this procedure.

12.3.3 Procedure for Preparing Laboratory Control Sample

12.3.3.1 For water samples, measure out 1.0 L of reagent water and spike with 1 mL of the LCS spiking solution (Section 7.2.2.6) and 1 mL of the surrogate standard spiking solution (Section 7.2.2.4). Extract, concentrate, and analyze the sample according to Section 10.0.

12.3.3.2 For soil/sediment samples, measure out 30 g of a clean reference matrix (e.g., sodium sulfate, Hydromatrix™) and spike with 1 mL of the LCS spiking solution (Section 7.2.2.6) and 1 mL of surrogate standard spiking solution (Section 7.2.2.4). Extract, concentrate, and analyze the LCS according to Section 10.0.

12.3.4 Calculations for Laboratory Control Sample

12.3.4.1 Calculate the results according to Section 11.0.

12.3.4.2 Calculate individual analyte recoveries of the LCS using Equation 13.

12.3.5 Technical Acceptance Criteria for Laboratory Control Sample

12.3.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

12.3.5.2 The LCS must be analyzed at the frequency described in Section 12.3.2 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.3.5.3 The LCS must be prepared as described in Section 12.3.3.

12.3.5.4 The LCS must meet all sample technical acceptance criteria in Sections 11.3.5 and 11.3.6.

12.3.5.5 The %R for each of the compounds in the LCS must be within the recovery limits listed in Table 8 – Laboratory Control Sample Recovery Limits.

12.3.5.6 Surrogate recoveries must fall within the acceptable criteria in Table 6 – Surrogate Recovery Limits. These limits are not advisory.

12.3.6 Corrective Action for Laboratory Control Sample

12.3.6.1 If the LCS technical acceptance criteria for the surrogates or the LCS compound recovery are not met, check calculations, the surrogate and LCS solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and LCS recovery criteria.

12.3.6.2 LCS technical acceptance criteria MUST be met before data are reported. LCS contamination from laboratory sources or any LCS analyzed not meeting the technical acceptance criteria will require re-extraction and reanalysis of the LCS at no additional cost to the EPA.

12.3.6.3 All samples (including MS/MSDs and PE samples) and required blanks, prepared and analyzed in an SDG with an LCS that does not meet the technical acceptance criteria, will also require re-extraction and reanalysis at no additional cost to the EPA.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed under the contract, the MDL for each Aroclor target analyte shall be determined on each instrument used for analysis. MDL determination is matrix-specific and level-specific (i.e., the MDL shall be determined for water and soil/sediment samples). The MDLs must be determined annually thereafter or after major instrument maintenance. Major instrument maintenance includes, but is not limited to: cleaning or replacement of the detector. A new MDL study will not be required after changing the GC column, as long as the replacement has the same length, inner diameter, and stationary phase.

12.4.2 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.3 The determined concentration of the MDL must be less than the CRQL listed in Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits.

12.4.4 All documentation for the MDL studies shall be maintained at the laboratory and submitted to the EPA within seven (7) days of study completion. This schedule and the designated recipients are specified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 of Exhibit D – Introduction to Organic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 of Exhibit D – Introduction to Organic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Automated Soxhlet Extraction, SW-846 Method 3541, Revision 0, September 1994.

16.2 U.S. Environmental Protection Agency, Continuous Liquid-Liquid Extraction, SW-846 Method 3520C, Revision 3, December 1996.

16.3 U.S. Environmental Protection Agency, Gel-Permeation Cleanup, SW-846 Method 3640A, Revision 1, September 1994.

16.4 U.S. Environmental Protection Agency, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, SW-846 Method 8082A, Revision 1, February 2007.

16.5 U.S. Environmental Protection Agency, Pressurized Fluid Extraction (PFE), SW-846 Method 3545A, Revision 1, February 2007.

16.6 U.S. Environmental Protection Agency, Separatory Funnel Liquid-Liquid Extraction, SW-846 Method 3510C, Revision 3, December 1996.

16.7 U.S. Environmental Protection Agency, Silica Gel Cleanup, SW-846 Method 3630C, Revision 3, December 1996.

16.8 U.S. Environmental Protection Agency, Sulfuric Acid/Permanganate Cleanup, SW-846 Method 3665A, Revision 1, December 1996.

16.9 U.S. Environmental Protection Agency, Ultrasonic Extraction, SW-846 Method 3550C, Revision 3, February 2007.

16.10 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. EPA REGISTRY NAMES, SYNONYMS, AND CHEMISTRY ABSTRACT SERVICE (CAS) REGISTRY NUMBERS

Systematic Name	EPA Registry Name	Synonym	CAS #
Aroclor 1016	Aroclor 1016	PCB-1016	12674-11-2
Aroclor 1221	Aroclor 1221	PCB-1221	11104-28-2
Aroclor 1232	Aroclor 1232	PCB-1232	11141-16-5
PCB 1242	Aroclor 1242	PCB-1242	53469-21-9
PCB 1248	Aroclor 1248	PCB-1248	12672-29-6
PCB 1254	Aroclor 1254	PCB-1254	11097-69-1
PCB 1260	Aroclor 1260	PCB-1260	11096-82-5
Aroclor 1262	Aroclor 1262	PCB-1262	37324-23-5
Aroclor 1268	Aroclor 1268	PCB-1268	11100-14-4
Benzene 1,2,3,5-tetrachloro-4,6-dimethyl	Tetrachloro-m-xylene	2,4,5,6-Tetrachloroxylene	877-09-8
1,1'-Biphenyl, 2,2',3,3',4,4',5,5',6,6'-decachloro	Decachlorobiphenyl	Decachloro-1,1'-biphenyl	2051-24-3

TABLE 2. CONCENTRATION LEVELS OF INITIAL CALIBRATION AND CONTINUING CALIBRATION VERIFICATION STANDARDS AND TECHNICAL ACCEPTANCE CRITERIA FOR AROCLORS

Analyte	Concentration (ng/mL)					Maximum %RSD	Opening Maximum %D	Closing Maximum %D
	CS1	CS2	CS3	CS4	CS5			
Aroclor 1016	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1221	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1232	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1242	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1248	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1254	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1260	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1262	100	200	400	800	1600	20.0	±25.0	±50.0
Aroclor 1268	100	200	400	800	1600	20.0	±25.0	±50.0
*Tetracholor-m-xylene	5.0	10.	20.	40.	80.	20.0	±30.0	±50.0
*Decachlorobiphenyl	10.	20.	40.	80.	160	20.0	±30.0	±50.0

*Surrogates are present in all calibration standards at the above concentrations.

NOTE: Aroclor 1016 and 1260 standards may be prepared together but the other Aroclor standards (1221 - 1268) must be prepared individually. For example, Aroclor 1016/1260 CS3 standard will contain both Aroclor 1016 and Aroclor 1260 at a concentration of 400 ng/mL, and the surrogates tetrachloro-m-xylene and decachlorobiphenyl at concentrations of 20 and 40 ng/mL, respectively. Aroclor 1242 CS1 Standard will contain only Aroclor 1242, tetrachloro-m-xylene, and decachlorobiphenyl at 100, 20, and 40 ng/mL, respectively.

TABLE 3. RETENTION TIME WINDOWS FOR ANALYTES AND SURROGATES

Compound	**Retention Time Windows (minutes)**
Aroclors	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

TABLE 4. GAS CHROMATOGRAPH ANALYTICAL CONDITIONS

Carrier Gas:	Helium or Hydrogen 99.999% purity
Column Flow:	5 mL/min.
Make-up Gas:	Argon/Methane (P-5 or P-10) or N_2 (required)
Injector Temperature:	> 200°C
Injection Technique:	On-column
Injection Volume:	1 or 2 µl
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	0.5 min.
Temperature Ramp:	5°C to 6°C/min.
Final Temperature:	275°C
Final Hold Time:	After decachlorobiphenyl has eluted

TABLE 5. CONCENTRATION OF MATRIX SPIKE/MATRIX SPIKE DUPLICATE SPIKING, LABORATORY CONTROL SAMPLE SPIKING, AND GEL PERMEATION CHROMATOGRAPHY CALIBRATION VERIFICATION STANDARD SOLUTIONS

Analyte	**MS/MSD Spiking Solution** (µg/mL)	**LCS Spiking Solution** (µg/mL)	**GPC Calibration Verification Solution** (µg/mL)
Aroclor 1016	4.0	1.0	0.40
Aroclor 1260	4.0	1.0	0.40

TABLE 6. SURROGATE RECOVERY LIMITS

Compound	Percent Recovery QC Limits
Tetrachloro-m-xylene	30-150
Decachlorobiphenyl	30-150

TABLE 7. MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Analyte	Percent Recovery Water/Soil	RPD Water/Soil
Aroclor 1016	29-135	0-15
Aroclor 1260	29-135	0-20

TABLE 8. LABORATORY CONTROL SAMPLE RECOVERY LIMITS

Analyte	Percent Recovery Water/Soil
Aroclor 1016	50-150
Aroclor 1260	50-150

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EXHIBIT E

QUALITY SYSTEMS

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Exhibit E - Quality Systems

Table of Contents

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1.0 QUALITY SYSTEM

1.1 Overview

Since the purpose of this analytical service is to provide analytical data for the use by the U.S. Environmental Protection Agency (EPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act (SARA), the Contractor is responsible for developing and implementing a Quality System to enforce the requirements of the EPA CIO 2105.0 "***Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs***". This will require the implementation of a quality system that meets the EPA's goal of providing data of documented quality.

1.1.1 The quality system provides the framework for planning, implementing, assessing, and improving work performed by the Contractor for performing quality assurance (QA) and quality control (QC) activities. Effective implementation of the quality system leads to several benefits including:

- Scientific Data Integrity – The Contractor will produce and submit data of known and documented quality;
- Effective Management of Internal and External Activities – The quality system requires documentation of activities and oversight for evaluation purposes which will reduce the potential for waste and abuse; and
- Continual Improvement – The continual improvement component of the quality system leads to the development of a better more responsive quality system and technical system which should result in better products and services.

1.1.2 Overall, successful implementation of the quality system will reduce the Agency's vulnerabilities in decision making and increase the EPA's credibility by providing the ability to make reliable, timely, cost effective, and defensible decisions. The consequences of not having a successfully implemented quality system include the potential to waste time, money, and resources, which increase uncertainty in the EPA's decision.

1.1.3 Under this program, the EPA requires two forms of documentation for the quality system:

- A Quality Management Plan (QMP) which documents the organization quality system; and
- A Quality Assurance Project Plan (QAPP) which documents the application of quality related activities to an activity-specific effort.

NOTE: The Contractor may combine these two documents into a single document that describes the organization's quality system and the application of this system to the work performed under this program.

2.0 QUALITY MANAGEMENT PLAN

During the contract solicitation process, the Contractor is required to submit the QMP or equivalent to the EPA Contracting Officer (CO). The QMP documents how an organization structures its quality system and describes its quality policies and procedures; criteria for and areas of application; and roles, responsibilities, and authorities. It also describes an organization's policies and procedures for implementing and assessing the effectiveness of the quality system. The Contractor shall follow the EPA Requirements for Quality Management Plans (QA/R-2) EPA/240/B-01/002 (or subsequent version) for guidance.

2.1 The QMP should describe the Quality System that is designed to support the objectives of the organization in providing the analytical services required in this document.

2.2 The QMP must be sufficiently inclusive, explicit, and readable to enable both management and staff to understand the priority which management places on QA and QC activities, established quality policies and procedures, and their respective quality related roles and responsibilities.

2.3 The QMP should document management practices, including QA and QC activities, used to ensure that the results of technical work are of the type and quality needed for their intended use.

2.4 The QMP should document the following: the mission and quality policy of the organization; the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities; the means by which effective communications with personnel actually performing the work are assured; the processes used to plan, implement, and assess the work performed; the process by which measures of effectiveness for QA and QC activities will be established and how frequently effectiveness will be measured; and the continual improvement based on lessons learned from previous experience.

2.5 The elements to be addressed in a QMP include: management and organization; quality system description; personnel qualifications and training; procurement of items and services; documentation and records; computer hardware and software; planning; implementation of work processes; assessment and response; and quality improvement.

NOTE: It is not necessary for the Contractor to present the information in the same order as outlined above as long as each item is adequately addressed in the plan.

3.0 QUALITY ASSURANCE PROJECT PLAN

3.1 Introduction

The EPA requires that all environmental data used in decision making be supported by an approved QAPP. The QAPP integrates all technical and quality aspects of a project including planning, implementation, and assessment. The purpose of the QAPP is to document how QA and QC are applied to an environmental data operation to assure that the results obtained are of the type and quality needed and expected for this program. The Contractor shall follow the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA/240/B-01/003) (or subsequent version) for guidance.

3.1.1 The Contractor shall prepare a written QAPP which describes the procedures that are implemented to:

- Maintain data integrity, validity, and usability;
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility;
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable; and
- Document all aspects of the measurement process to provide data which are technically sound and legally defensible.

3.1.2 The QAPP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAPP.

3.1.3 The QAPP shall be available during on-site laboratory evaluations.

3.1.4 The QAPP shall be submitted within 7 days of written request by the EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR) or the Analytical Services Branch CLP COR (ASB CLP COR).

3.2 Required Elements of a Quality Assurance Project Plan

The QAPP shall be paginated consecutively in ascending order. The required elements of a laboratory's QAPP are outlined in this section. This outline should be used as a framework for developing the QAPP.

A. Organization and Personnel

1. QA Policy and Objectives (the mission and quality policy of the organization)
2. QA Management (the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities)
    a. Organization
    b. Assignment of QA/QC Responsibilities
    c. Reporting Relationships (the means by which effective communication with personnel actually performing the work are ensured)
    d. QA Document Control Procedures

e. QA Program Assessment Procedures (the process used to plan, implement, and assess the work performed)

3. Key Personnel (laboratory personnel involved in QA and QC activities)

a. Resumes

b. Education and Experience Relevant to this Contract

c. Training Records and Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives

2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy

2. Sample Tracking/Custody Procedures

3. Logbook Maintenance and Archiving Procedures

4. Complete Sample Delivery Group (SDG) File (CSF) Organization, Preparation, and Review Procedures

5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs

6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency

2. Sample Preparation/Extraction Procedures

3. Sample Analysis Procedures

4. Standards Preparation Procedures

5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures

2. Data Reduction Procedures

3. Data Validation Procedures

4. Data Reporting and Authorization Procedures

F. QA (the process which measures the effectiveness of QA will be established and how frequently effectiveness will be measured)

1. Data QA

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures (the continual improvement based on lessons learned from previous experience)

5. QA Reporting Procedures

6. Responsibility Designation

G. QC

1. Solvent, Reagent, and Adsorbent Check Analysis
2. Reference Material Analysis
3. Internal QC Checks
4. Corrective Action and Determination of QC Limit Procedures
5. Responsibility Designation

3.3 Submission of the Quality Assurance Project Plan

3.3.1 Initial Submission

The Contractor is required to submit their QAPP to the EPA CO within the number of days provided in the associated laboratory contract document. The Contractor shall maintain a QAPP (fully compliant with the requirements of this contract) on file at their facility for the term of the contract.

3.3.2 Revision Submissions

The revised QAPP will become the official QAPP under the contract and may be used during legal proceedings.

3.3.2.1 During the term of the contract, the Contractor shall amend the QAPP when the following circumstances occur:

- The EPA modifies technical requirements of the Statement of Work (SOW) or the contract;
- The EPA notifies the Contractor of deficiencies in the QAPP document;
- The EPA notifies the Contractor of deficiencies resulting from the EPA's review of the Contractor's performance;
- The Contractor identifies changes in organization, personnel, facility, equipment, policy, or procedures; or
- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, procedure, or QAPP document.

3.3.2.2 The Contractor shall amend and submit the QAPP to the recipient(s) identified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule, within 14 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAPP, and what is presently occurring at the Contractor's facility.

3.3.2.2.1 All changes in the QAPP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font) and the amended section pages shall have the date on which the changes were implemented.

3.3.2.2.2 The Contractor shall archive all amendments to the QAPP document for future reference by the Government.

3.3.2.3 The Contractor shall send a copy of the latest version of the QAPP document within 7 days of a written request by the EPA Regional CLP COR or the ASB CLP COR, as directed. The EPA requestor will designate the recipients.

4.0 STANDARD OPERATING PROCEDURES

4.1 Introduction

To obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of SOPs. As defined by the EPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. The Contractor shall follow the EPA Guidance for Preparing Standard Operating Procedures (SOPs) (QA/G-6).

4.1.1 SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up to date, and sufficiently detailed to permit duplication of results by qualified analysts).

4.1.2 All SOPs shall reflect activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current EPA regulations, guidelines, and the CLP contract's requirements;
- Consistent with instrument(s) manufacturer's specific instruction manuals;
- Available to the Government during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs;
- Available to designated recipients within 7 days, upon request by the EPA Regional CLP COR or the ASB CLP COR;
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol;
- Capable of demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results;
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements;
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made;
- Archived for future reference in usability or evidentiary situations;
- Available at specific workstations, as appropriate;
- Reviewed and signed by all Contractor personnel performing actions identified in the SOP; and
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

4.2 Format

The format for SOPs may vary depending upon the type of activity for which they are prepared. The SOPs shall be paginated consecutively in ascending order. At a minimum, the following sections shall be included:

- Title Page;
- Document Control;
- Scope and Applicability;
- Summary of Method;
- Definitions (acronyms, abbreviations, and specialized forms used in the SOP);
- Health and Safety;
- Personnel Qualifications;
- Interferences;
- Apparatus and Materials (list or specify, also note designated locations where found);
- Handling and Preservation;
- Instrument or Method Calibration;
- Sample Preparation and Analysis;
- Data Calculations;
- Procedures;
- QC limits;
- Corrective action procedures, including procedures for secondary review of information being generated;
- Documentation description and example forms;
- Data Management and Records Management;
- Miscellaneous notes and precautions; and
- References.

4.3 Required Standard Operating Procedures

The Contractor shall maintain the following SOPs:

4.3.1 Evidentiary SOPs for required chain of custody and document control.

4.3.2 Sample receipt and storage:

- Sample receipt and identification logbooks;
- Refrigerator temperature logbooks;
- Extract storage logbooks; and
- Security precautions.

4.3.3 Sample preparation:

- Reagent purity check procedures and documentation;
- Extraction procedures;
- Extraction bench sheets; and
- Extraction logbook maintenance.

4.3.4 Glassware cleaning

4.3.5 Calibration (balances, pipets, etc.):

- Procedures;
- Frequency requirements;
- Preventative maintenance schedule and procedures;
- Acceptance criteria and corrective actions; and
- Logbook maintenance authorization.

4.3.6 Analytical procedures (for each analytical system):

- Instrument performance specifications;
- Instrument operating procedures;
- Data acquisition system operation;
- Procedures used when automatic quantitation algorithms are overridden;
- QC-required parameters;
- Analytical sequence/injection logbooks; and
- Instrument error and editing flag descriptions and resulting corrective actions.

4.3.7 Maintenance activities (for each analytical system):

- Preventative maintenance schedule and procedures;
- Corrective maintenance determinants and procedures; and
- Maintenance authorization.

4.3.8 Analytical standards:

- Standard coding/identification and inventory system;
- Standards preparation logbook(s);
- Standard preparation procedures;
- Procedures for equivalency/traceability analyses and documentation;
- Purity logbook (primary standards and solvents);
- Storage, replacement, and labeling requirements; and
- QC and corrective action measures.

4.3.9 Data reduction procedures:

- Data processing systems operation;
- Outlier identification methods;

- Identification of data requiring corrective action; and
- Procedures for format and/or forms for each operation.

4.3.10 Documentation policy/procedures:

- Contractor/analyst's notebook policy, including review policy;
- CSF contents;
- CSF organization and assembly procedures, including review policy; and
- Document inventory procedures, including review policy.

4.3.11 Data validation/self-inspection procedures:

- Data flow and chain of command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to ensure that hardcopy and electronic deliverables are complete and compliant with the requirements in Exhibit B – Reporting and Deliverables Requirements and Exhibit H – Format for Electronic Data Deliverables;
- Procedures to ensure that hardcopy deliverables are in agreement with their comparable electronic deliverables;
- Demonstration of internal QA inspection procedure [demonstrated by supervisory sign-off on personal notebooks, internal Performance Evaluation (PE) samples, etc.];
- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

4.3.12 Data management and handling:

- Procedures for controlling and estimating data entry errors;
- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates;
- Lifecycle management procedures for testing, modifying, and implementing changes to existing computing systems to include hardware, software, and documentation or installation of new systems;
- Database security, backup, and archival procedures including recovery from system failures;
- System maintenance procedures and response time;
- Individual(s) responsible for system operation, maintenance, data integrity, and security;
- Specifications for staff training procedures;

- Virus Protection procedures for software and electronic data deliverables; and
- Storage, retrieval, and verification of the completeness and readability of instrument files transferred to electronic media.

4.4 Submission of the Standard Operating Procedures

4.4.1 Initial Submission

The Contractor is required to submit their SOPs to the EPA CO within 60 days after contract award. The Contractor shall maintain on file a complete set of SOPs, fully compliant with the requirements of this contract for the term of the contract.

4.4.2 Revision Submissions

The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings.

4.4.2.1 During the term of the contract, the Contractor shall amend the SOPs when the following circumstances occur:

- The EPA modifies the technical requirements of the SOW or the contract;
- The EPA notifies the Contractor of deficiencies in their SOP documentation;
- The EPA notifies the Contractor of deficiencies resulting from the EPA's review of the Contractor's performance;
- The Contractor's procedures change;
- The Contractor identifies deficiencies resulting from the internal review of SOP documentation; or
- The Contractor identifies deficiencies resulting from the internal review of procedures.

4.4.2.2 The Contractor shall amend and submit revised or write and submit new SOPs to the recipient(s) identified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule, within 14 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs, and what is presently occurring at the Contractor's facility.

4.4.2.2.1 All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font) and the amended/new SOPs shall have the date on which the changes were implemented.

4.4.2.2.2 The Contractor shall document the reasons for the changes and archive all amended SOPs for future reference by the Government. Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs.

4.4.2.3 The Contractor shall send a copy of the latest version of the SOPs within 7 days of a written request by the EPA Regional CLP COR or the ASB CLP COR, as directed. The EPA requestor will designate the recipients.

5.0 CHAIN OF CUSTODY

5.1 Introduction

A sample is physical evidence collected from a facility or the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that the EPA's sample data and records supporting sample-related activities are admissible as evidence in litigation, Contractors are required to maintain EPA furnished samples under chain of custody and to account for all samples and supporting records of sample handling, preparation, and analysis.

The Contractor shall develop and implement the following SOPs for sample chain of custody (COC) under this contract. The Contractor shall provide the following SOPs: sample receiving, sample identification, sample security, sample storage, sample tracking and document control, electronic sample data control, and CSF organization and assembly to ensure accountability of sample chain of custody, as well as control of all sample-related records.

5.2 Sample Receiving

5.2.1 The Contractor shall designate a sample custodian responsible for receiving Government-furnished samples.

5.2.2 The Contractor shall designate a representative to receive Government-furnished samples in the event that the sample custodian is not available.

5.2.3 The sample custodian or a designated representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

5.2.4 The sample custodian or a designated representative shall verify and record the following information on Form DC-1 as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers, when present;
- Condition of the sample bottles;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;
- Presence or absence of Traffic Report/Chain of Custody (TR/COC) Records;
- Sample tags/numbers listed/not listed on TR/COC Records;
- Presence or absence of shipping container temperature indicator bottle;
- Shipping container temperature;
- Date of receipt;
- Time of receipt;
- EPA Sample Numbers;

- Presence or absence of sample tags;
- Sample tag numbers;
- Assigned laboratory numbers;
- Remarks regarding condition of sample shipment;
- Samples delivered by hand; and
- Problems and discrepancies.

5.2.5 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., TR/COC Records or packing lists, and airbills).

NOTE: Initials are not acceptable.

5.2.6 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to: absent documents, conflicting information, and absent or broken custody seals.

5.2.7 The Contractor shall record resolution of all problems and discrepancies communicated through SMO.

5.3 Sample Identification

5.3.1 The Contractor shall maintain the identity of Government-furnished samples and prepared samples (including extracts) throughout the laboratory.

5.3.2 Each sample and sample preparation container shall be labeled with the EPA Sample Number or a unique laboratory sample identification number.

5.4 Sample Security

5.4.1 The Contractor shall demonstrate that sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession; or
- It is in your view after being in your possession; or
- It is locked in a secure area after being in your possession; or
- It is in a designated secure area, accessible only to authorized personnel.

5.4.2 The Contractor shall demonstrate security of designated secure areas.

5.5 Sample Storage

The Contractor shall designate storage areas for Government-furnished samples and prepared samples.

5.6 Sample Tracking and Document Control

5.6.1 The Contractor shall record all activities performed on Government-furnished samples.

5.6.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents (activities include, but are not limited to: sample receipt, sample storage, sample preparation, sample analysis, CSF organization and assembly, and sample retention or disposal). When a document is a record of analysis, the instrument type and parameter group shall be included in the title.

5.6.3 When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

5.6.4 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

NOTE: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity. The Laboratory Name shall be identified on pre-printed laboratory documents.

5.6.5 Each laboratory document entry shall be dated in the format MM/DD/YYYY (e.g., 01/01/2016) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

5.6.6 Notations on laboratory documents shall be recorded in ink.

5.6.7 Corrections to 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.

5.6.8 Unused portions of laboratory documents shall be lined out, signed (or initialed), and dated.

5.6.9 Pages in bound and unbound logbooks shall be sequentially numbered.

5.6.10 Each page in bound and unbound logbooks shall be dated (MM/DD/YYYY) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page).

5.6.11 Instrument-specific analytical sequence logs shall be maintained to enable the reconstruction of analytical sequences.

5.6.12 Logbook entries shall be in chronological order.

5.6.13 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.

5.6.14 The Contractor shall document disposal or retention of Government-furnished samples, remaining portions of samples, and prepared samples.

5.7 Electronic Sample Data Control

5.7.1 Contractor personnel responsible for original data entry shall be identified at the time of data input.

5.7.2 The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.

5.7.3 The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.

5.7.4 The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.

5.7.5 The Contractor shall ensure that the electronic data collection system is secure.

5.7.5.1 The electronic data collection system shall be maintained in a secure location.

5.7.5.2 Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (e.g., log-ons or restricted passwords).

5.7.5.3 Electronic data collection systems shall be protected from the introduction of external programs or software (e.g., viruses).

5.7.6 The Contractor shall designate archive storage areas for electronic data and the software required to access the data.

5.7.7 The Contractor shall designate an individual responsible for maintaining archives of electronic data, including the software.

5.7.8 The Contractor shall maintain the archives of electronic data and necessary software in a secure location that shall be accessible only to authorized personnel.

5.8 Complete Sample Delivery Group File Organization and Assembly

5.8.1 The Contractor shall designate a Document Control Officer responsible for the organization and assembly of the CSF.

5.8.2 The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the Document Control Officer is not available.

5.8.3 The Contractor shall maintain documents relating to the CSF in a secure location.

5.8.4 All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.

5.8.5 Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.

5.8.6 Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF:

- Logbook pages;
- Bench sheets;
- Screening records;
- Preparation records;
- Repreparation records;
- PE sample instructions;
- Chromatograms;
- Analytical records;
- Reanalysis/Re-extraction records;
- TR/COC Records;

- Sample tracking records;
- Raw data summaries;
- Computer printouts;
- Records of failed or attempted analysis;
- Correspondence;
- FAX originals; and
- Other.

5.8.7 The Document Control Officer or a designated representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.

5.8.8 CSF documents shall be organized and assembled on an SDG-specific basis.

5.8.9 Original documents which include information relating to more than one SDG (e.g., TR/COC Records, calibration logs) shall be filed in the CSF with the lowest SDG Number, and copies of these originals shall be placed in the other CSF(s). The Document Control Officer or a designated representative shall record the following statement on the copies in (indelible) dark ***ink***:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF

_____
Signature

_____
Date

5.8.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.

5.8.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

5.8.12 Before shipping each CSF, the Document Control Officer or a designated representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

5.8.13 The Document Control Officer or a designated representative shall document the shipment of deliverable packages, including what was sent, to whom the packages were sent, the date, and the carrier used.

5.8.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the Document Control Officer or a designated representative in a manner such that opening the packages would break the seals.

5.8.15 Custody seals shall be signed and dated by the Document Control Officer or a designated representative when sealing deliverable packages.

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EXHIBIT F

PROGRAMMATIC QUALITY ASSURANCE/QUALITY CONTROL ELEMENTS

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Exhibit F – Programmatic Quality Assurance/Quality Control Elements

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1.0 OVERVIEW

Quality Assurance (QA) and Quality Control (QC) are integral parts of the U.S. Environmental Protection Agency's (EPA's) Contract Laboratory Program (CLP). This integrated program is required to generate data of known and documented quality. The QA process consists of management reviews and oversight at the planning, implementation, and completion stages of the environmental data collection activity, and ensures that data provided are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.

During the planning of an environmental data collection program, the activities focus on defining data quality criteria and designing a QC system to measure the quality of the data being generated. During the implementation of the data collection effort, the QA activities ensure that the QC system is functioning effectively, and the deficiencies uncovered by the QC system are corrected. After the environmental data are collected, QA activities focus on assessing the quality of data obtained to determine it suitability to support enforcement or remedial decisions.

2.0 INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the QC procedures and criteria incorporated into the methods. The data acquired from QC procedures are used to estimate and evaluate the information content of analytical results and to determine the necessity for, or the effects of, corrective action procedures. The parameters used to estimate information content include precision, accuracy, and other quantitative and qualitative indicators.

This Exhibit describes the overall programmatic QA/QC operations and the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing the EPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

3.0 GENERAL QUALITY ASSURANCE/QUALITY CONTROL PRACTICES

The necessary components of a complete QA/QC program include internal QC criteria that demonstrate compliant levels of performance, as determined by the Contractors' QA review and external QC review of data and procedures that is accomplished by the monitoring activities of the EPA.

Each external review accomplishes a different purpose. External reviews may include: Proficiency Testing, contract compliance screening, on-site laboratory audits, data package audits, electronic data audits, and the EPA Regional data review. A feedback loop provides the results of these various review functions to the Contractor through communications with the EPA.

4.0 PROFICIENCY TESTING PROGRAM

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor shall participate in the EPA's Proficiency Testing (PT) Program. The EPA's PT Program involves the analysis of Case-specific Performance Evaluation (PE) samples and PT audits. The Contractor's PE and PT audit sample results will be used by the EPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements. The Contractor must receive a passing score of 75% to be in compliance with the contract.

4.1 Performance Evaluation Samples

4.1.1 PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis.

4.1.2 PE samples will be provided as either single-blinds (recognizable as a PE sample, but of unknown composition), or as double-blinds (not recognizable as a PE sample and of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PE samples.

4.1.3 The Contractor may receive the PE samples as either full volume samples or ampulated/bottled concentrates from the EPA or a designated EPA Contractor. The PE samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PE samples (i.e., the required dilution of the PE sample concentrate). PE samples are to be extracted and analyzed with the rest of the routine samples in the SDG. The Contractor shall prepare and analyze the PE sample using the procedure described in the sample preparation and method analysis sections of Exhibit D – Analytical Methods. All contract required QC shall also be met.

4.1.4 The PE sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B – Reporting and Deliverables Requirements. If these requirements are not met, the EPA Region may reject all the data associated with the SDG.

4.1.5 The Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PE sample. When PE sample results are received by the EPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The results of the PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte. The EPA will notify the Contractor of unacceptable performance.

4.2 Proficiency Testing Audits

4.2.1 A PT audit is a unique analytical Case containing only PT audit samples. The PT audit samples will be scheduled by the EPA Analytical Services Branch (ASB) through the Sample Management Office (SMO). PT audit samples assist the EPA in monitoring Contractor performance.

4.2.2 PT audit samples will be provided as single-blinds (recognizable as a PT audit sample but of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PT audit samples.

4.2.3 The Contractor may receive the PT audit samples as either full volume samples or ampulated/bottled concentrates from the EPA or a designated EPA Contractor. The PT audit samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PT audit samples (i.e., the required dilution of the PT audit sample concentrate). The Contractor shall prepare and analyze the PT audit samples using the procedure described in the sample preparation and method analysis sections of Exhibit D – Analytical Methods. All contract required QC shall be met, including spike and spike duplicate.

4.2.4 The PT audit sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B - Reporting and Deliverables Requirements.

4.2.5 The Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PT audit sample. When PT audit sample results are received by the EPA, the PT audit sample results will be scored for correct analytical identification, quantitation, and timeliness. The PT audit sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte.

4.2.6 The EPA will notify the Contractor of unacceptable performance. The Contractor's overall and method-specific PT audit sample performance will be assessed into one of the following three categories:

4.2.6.1 Acceptable, No Response Required: Score greater than or equal to 90%. The data meets most or all of the scoring criteria. No response is required.

4.2.6.2 Acceptable, Response Explaining Deficiencies Required: Score greater than or equal to 75%, but less than 90%. Deficiencies exist in the Contractor's performance. Corrective action response required.

4.2.6.3 Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75%. Corrective action response required.

4.2.7 In the case of Section 4.2.6.2 or 4.2.6.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the EPA Contracting Officer (CO), the EPA Regional CLP Contracting Officer's Representative (COR), and the ASB CLP COR, within 14 days of receipt of notification from the EPA.

4.2.8 A remedial PT audit is a unique analytical Case containing only PT audit samples. A remedial PT audit may be scheduled by EPA ASB with the Contractor(s) for any of the following reasons: unacceptable PE sample performance and/or major change in the laboratory (e.g., relocation, new owner, or high turnover of key personnel). The Contractor may not receive samples under this contract until acceptable performance of a remedial PT audit sample is achieved. Sections 4.2.2 through 4.2.7 apply to the remedial PT audit process.

4.2.9 The Contractor shall be notified by the EPA CO concerning agreement or disagreement with the proposed remedy for unacceptable performance.

5.0 CONTRACT COMPLIANCE SCREENING

5.1 Overview

5.1.1 Contract Compliance Screening (CCS) is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the Complete SDG File (CSF) delivered to the EPA.

5.1.2 CCS is performed by SMO at the direction of the EPA. To ensure uniform review, a set of standardized procedures has been developed to evaluate the CSF submitted by a Contractor against the technical and completeness requirements of the contract. The EPA reserves the right to add and/or delete individual checks.

5.2 Contract Compliance Screening Results

CCS results are distributed to the Contractor and all other data recipients. The Contractor shall correct deficiencies and submit corrections within 6 business days. The Contractor shall send all corrections to the EPA Regional CLP COR and SMO. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

5.3 Contract Compliance Screening Trend Report

The EPA will periodically generate a CCS Trend Report which summarizes CCS results over a given period of time. The Government may send the CCS Trend Report to the Contractor, or discuss the CCS Trend Report during an on-site laboratory audit. In a detailed letter to the EPA Regional CLP COR, the ASB CLP COR, and the EPA CO, the Contractor shall address the deficiencies and the subsequent corrective actions implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report.

6.0 ON-SITE LABORATORY AUDITS

6.1 Overview

The EPA Regional CLP COR, the ASB CLP COR, or the EPA CO's authorized representative will conduct an on-site laboratory audit. On-site laboratory audits are performed to monitor the Contractor's ability to meet selected terms and conditions specified in the contract.

6.2 On-Site Audit

QA evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation; the continuity, experience and education of personnel; and the acceptable performance of analytical and QC procedures. Auditors conduct on-site laboratory audits to evaluate if laboratory policies and procedures are in place to satisfy evidence handling requirements.

6.2.1 The items to be monitored during an on-site audit may include, but not be limited to, the following:

- Size and appearance (e.g., cleanliness, organization) of the facility;
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;
- Availability, review, appropriateness, and utilization of the Quality Assurance Project Plan (QAPP) and Standard Operating Procedures (SOPs);
- Staff qualifications, experience, and personnel training programs;
- Analysis of PE samples (may be in the presence of the EPA-designated team);
- Reagents, standards, and sample storage facilities;
- All logbooks (e.g., extraction logs, standards and reagent preparation logs, analysis logs, instrument maintenance logs);
- All raw analytical data; and
- Review of the Contractor's sample analysis, data package assembly, inspection, completion, and data management procedures.

6.2.2 Prior to an on-site audit, various documentation pertaining to performance of the Contractor is reviewed by the audit team and may be discussed during the audit. Items that may be discussed include, but not limited to, the following:

- Previous on-site audit reports;
- PE or PT audit sample scores;
- EPA Regional review of data;
- Contractor performance information;
- Data and Electronic audit reports;
- Results of CCS; and
- Data trend reports.

6.3 Discussion of the On-Site Audit Findings

The auditors shall present their findings and recommendations for corrective actions necessary to the Contractor personnel during a debriefing meeting at the conclusion of the audit. A report which discusses deficiencies found during the on-site audit will be sent to the Contractor to provide further clarification of findings.

6.3.1 In a detailed letter to the EPA Regional CLP COR, the ASB CLP COR, and the EPA CO, the Contractor shall discuss the deficiencies and the subsequent corrective actions implemented by the Contractor to resolve the deficiencies within 14 days of receipt of report.

7.0 DATA PACKAGE AUDITS

7.1 Overview

Audits provide the EPA with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. Data package audits enable the EPA to evaluate the implementation, precision, and accuracy of the analytical methods. The audits are performed by the EPA to support the following activities:

- Program overview;
- Contractual requirements and data consistency;
- Identification/Investigation of data quality problems;
- Support for on-site laboratory audits; and
- Specific EPA Regional requests.

7.2 Required Information

Data packages are periodically selected from recently received Cases and evaluated for the technical quality of hardcopy raw data, QA, and the adherence to contractual requirements. A thorough review of the raw data is completed, including all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements; a check for transcription and calculation errors; a review of the qualifications of the laboratory personnel involved with the Case; and a review of the latest version of all SOPs on file. This function provides external monitoring of program QC requirements. Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance.

7.3 Submission Request

The data package from a recent Case, a specific Case, or a PE sample may be requested. Upon request from the EPA Regional CLP COR, the ASB CLP COR, or the EPA CO, the Contractor shall send the required data package and all necessary documentation to the EPA designated recipient within 7 days of notification in accordance with Exhibit B - Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

7.4 Response to the Data Package Audit Report

After completion of the data package audit, the EPA shall make the data package audit report available to the Contractor. In a detailed letter to the designated recipients, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

8.0 ELECTRONIC DATA AUDITS

8.1 Overview

Audits provide the EPA with an in-depth inspection and evaluation of the electronic data with regard to achieving QA/QC acceptability. Electronic data audits enable the EPA to evaluate the implementation, precision, and accuracy of the analytical methods. The audits are performed by the EPA to support the following activities:

- Program overview;
- Contractual requirements and data consistency;
- Identification/Investigation of data quality problems;
- Support for on-site laboratory audits; and
- Specific EPA Regional requests.

8.2 Required Information

Data packages are periodically selected from recently received Cases and evaluated for the technical quality of hardcopy raw data, QA, and the adherence to contractual requirements. A thorough review of the raw data is completed, including all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements; a check for transcription and calculation errors; a review of the qualifications of the laboratory personnel involved with the Case; and a review of the latest version of all SOPs on file. This function provides external monitoring of program QC requirements. Electronic data audits are used to assess the technical quality of the data and evaluate overall laboratory performance.

8.2.1 The Contractor shall store all raw and processed analytical data in appropriate instrument manufacturer's proprietary software format uncompressed and with no security codes. This data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. The Contractor is required to retain the instrument electronic data for 3 years after submission of the reconciled CSF.

8.2.2 All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This electronic data shall include data for all samples, blanks, Laboratory Control Samples (LCSs), matrix spikes and matrix spike duplicates, tunes, initial calibrations, initial calibration verifications, and continuing calibration verifications.

8.2.3 The Contractor shall maintain a written reference logbook of data files of the EPA Sample Number, calibration data, standards, spikes, duplicates, and blanks. The logbook shall include the EPA Sample Numbers and standard and blank IDs, identified by Case.

8.2.4 The Contractor shall supply upon request raw data for the Method Detection Limit (MDL) studies which are used to set the MDL values for the SDG.

8.2.5 Electronic data shipped to the EPA-designated recipient must be fully usable by the recipient. When submitting instrument electronic data to the EPA, the following materials shall be delivered in response to the request:

8.2.5.1 All associated raw data files for all analytical samples, calibration, and QC data.

8.2.5.2 All processed data files and quantitation output files associated with the raw data files described in Section 8.2.5.1.

8.2.5.3 All associated identification and calculation files used to generate the data submitted in the data package. This includes, but is not limited to: result files, acquisition files, calibration files, and method files.

8.2.5.4 References relating data files to EPA Sample Numbers, calibration data, standards, blanks, spikes, and LCSs. The logbook shall include the EPA Sample Numbers and Lab File Identifiers for all samples, blanks, and standards, identified by Case and SDG.

8.2.5.5 A printout of the directory of all files in each directory, including all subdirectories and the files contained therein.

8.2.5.6 A copy of the CSF, if an audit request is made within the period during which the Contractor must retain a copy.

8.2.5.7 A statement attesting to the completeness of the instrument electronic data submission, signed and dated by the Contractor's Laboratory Manager or Manager's designee. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a cover sheet that includes the following information relevant to the data file submission:

- Contractor name;
- Date of submission;
- Case Number;
- SDG Number;
- Instrument manufacturer and model number;
- Instrument operating software and version number;
- Data system computer;
- System operating software;
- Data system network;
- Data backup software/service;
- Data analysis software;
- Media type and volume of data (in MB) backed up; and
- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

8.3 Submission of Request

The instrument electronic data from a recent Case, a specific Case, or a PE sample may be requested. Upon request from the EPA Regional CLP COR, the ASB CLP COR, or the EPA CO, the Contractor shall send the required instrument electronic data and all necessary documentation to the EPA designated recipient within 7 days of notification in accordance with Exhibit B - Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

8.4 Response to the Electronic Data Audit Report

After completion of the electronic data audit, the EPA will make the electronic data audit report available to the Contractor. In a detailed letter to the designated recipients, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the electronic data audit report within 14 days of receipt of the report.

9.0 REGIONAL DATA REVIEW

9.1 Overview

Contractor data are generated to meet the specific needs of the EPA Regions. In order to verify the usability of data for the intended purpose, each EPA Region reviews data from the perspective of the end user, based on functional guidelines for data review, which have been developed jointly by the EPA Regions and EPA ASB. Each EPA Region uses the guidelines as the basis for data evaluation. Individual EPA Regions may augment the basic guideline review process with additional review based on the EPA Region-specific or site-specific concerns. The EPA Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the EPA Regional response appropriate to the specific circumstances.

The EPA Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done by SMO, which is designed to identify contractual discrepancies, and the review done by EPA ASB, which is designed to evaluate Contractor and method performance.

9.2 Submission Request

As part of the CLP contractual requirements, CLP laboratories shall deliver their CSF for each SDG to the EPA Region where the samples have been collected. The EPA Regional recipients are identified at the time of scheduling. The data shall be shipped in accordance to the procedures described in Exhibit B - Reporting and Deliverables Requirements of this Statement of Work (SOW). The EPA Regions use the hardcopy data to perform their data review. The EPA Region may contact the laboratory after they initiate or complete their review requesting additional information or clarification. The Contractor shall respond to the request within 5 business days (exception 3 days for a 7-day turnaround).

10.0 TABLES

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit A: Summary of Requirements	Summary of Program Requirements	Performance standards are summarized in Exhibit A, Sections 1.0 through 4.0.	QA monitoring plan is outlined in Exhibit F.
Exhibit B: Reporting and Deliverables Requirements	Reporting and Deliverable Requirements	Performance standards are outlined in Exhibit B, Sections 1.0 through 4.0.	CCS in Exhibit F, Section 5.0, and SMO data review will be used to monitor reporting electronic deliverables.
Exhibit C: Organic Target Analyte List and Contract Required Quantitation Limits	Target Analyte List and Contract Required Quantitation Limits	Performance standards are outlined in Exhibit C.	QA monitoring plan is outlined in Exhibit F.
Exhibit D: Organic Analytical Methods	Introduction to Analytical Methods	Performance standards for stock standards are outlined in Exhibit D, Introduction, Section 4.0, and must be performed as stated.	Randomly, the EPA will review analytical standards verification and preparation documentation, as deemed appropriate.
	General Organic Analyses requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	Trace Volatiles requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	Low/Medium Volatiles requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit D: Organic Analytical Methods (Cont'd)	Semivolatiles requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	Pesticides requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	Aroclors requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
Exhibit E: Quality Systems	General QA/QC Requirements	As outlined in each Exhibit D, Section 12.0.	QA Management Plan is outlined in Exhibit E, Section 2.0.
	Quality Assurance Project Plan	As outlined in Exhibit E, Section 3.0, a written QAPP shall be used to ensure acceptable data production of known and documented quality.	The EPA will review and approve the QAPP after contract award and throughout the contract term as needed. *[The Quality Management Plan (QMP) will be reviewed and approved by the EPA pre contract award.]*
	Standard Operating Procedures	Performance standards are outlined in Exhibit E, Section 4.0, and must be performed as stated.	SOPs will be reviewed by the EPA during on-site audits, after modifications are made, and randomly, as deemed appropriate.
	Data Management	Performance standards are outlined in Exhibit E, Section 4.3.12.	The EPA will monitor data management practices during quality assurance and evidentiary on-site audits.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit F: Programmatic Quality Assurance/ Quality Control Elements	Proficiency Audit Testing	Performance standards are outlined in Exhibit F, Section 4.0, and must be performed as stated.	Acceptable PT audit scores will assist in monitoring Contractor performance as defined in Exhibit F, Section 4.2.5.
	Contract Compliance Screening	Performance standards are outlined in the contract and must be performed as stated.	CSF will be evaluated against the technical and completeness requirements of the contract.
	On-Site Laboratory Audits	Performance standards are outlined in Exhibit F, Section 6.2.	The EPA will evaluate the results from quality assurance and evidentiary on-site audits as defined in Exhibit F, Section 6.3, to assist in monitoring the Contractor.
	Data Package Audits	Performance standards are outlined in Exhibit F, Section 7.0.	Data package audits are performed by the EPA to evaluate technical quality of the hardcopy raw data, QA, and adherence to contractual requirements.
	Electronic Data Evaluation and Audits	Performance standards are outlined in Exhibit F, Section 8.0.	The EPA uses Exhibit F, Section 8.0, to monitor laboratory electronic deliverables.
	Regional Data Review	Analytical data is reviewed by each Region from the perspective of the end user to determine the usability of the data, as outlined in Exhibit F, Section 9.0.	The EPA Regional validation and/or SMO data review reports are generated for all data packages.
Exhibit G: Glossary of Terms	Glossary of Terms	Contractors shall adhere to interpretation of SOW terms as defined within Exhibit G.	N/A
Exhibit H: Format for Electronic Data Deliverables	Data Dictionary and Format	Performance standards are outlined in Exhibit H.	CCS in Exhibit F, Section 5.0, will be used to monitor electronic deliverables.

EXHIBIT G

GLOSSARY OF TERMS

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ALIQUOT - A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ALKANE - Any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the injection of the sample, standard, or blank into the Gas Chromatograph/Mass Spectrometer (GC/MS) or GC system.

ANALYTE – The specific compound an analysis seeks to determine.

ANALYTICAL METHOD - Specifies the procedures for sample preparation, instrument calibration, sample analysis, and result calculations.

ANALYTICAL REFERENCE STANDARD – Standards purchased from private chemical supply companies used to prepare calibration standards, Initial Calibration Verification (ICV) standards, and Continuing Calibration Verification (CCV) standards.

ANALYTICAL SAMPLE – Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), and tunes. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA); matrix spike samples; matrix spike duplicate samples; Laboratory Control Samples (LCSs); Performance Evaluation (PE) samples; and method, storage, cleanup, and Method Instrument Blanks.

ANALYTICAL SEQUENCE - The order of actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final Continuing Calibration Verification (CCV). All sample analyses during the analytical sequence are subject to the Quality Control (QC) protocols set forth in Exhibit D – Analytical Methods and Exhibit F – Programmatic Quality Assurance/Quality Control Elements of the contract, unless otherwise specified in the individual methods.

ANALYTICAL SERVICES BRANCH (ASB) - The division of the United States Environmental Protection Agency’s (EPA’s) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ASTM/ASTM INTERNATIONAL – A developer and provider of voluntary consensus standards.

BAR GRAPH SPECTRUM - A plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include calibration blanks, instrument blanks, method blanks, and field blanks. See the individual definitions for types of blanks.

BREAKDOWN - A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

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

CALIBRATED MASS – 1) A mass whose apparent mass has been adjusted from the uncalibrated mass by the instrumental mass calibration software routine. 2) An analyte mass whose intensity counts have been calibrated against standards of known analyte concentration.

CALIBRATION - A set of operations that establish under specific conditions, the relationship between values indicated by a measuring instrument and the corresponding known values.

CALIBRATION FACTOR (CF) - A measure of the Gas Chromatographic response of a target analyte to the mass injected.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the calibration curve). The solutions may or may not be subjected to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

CASE - A finite, usually predetermined number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

CHARACTERIZATION - A determination of the approximate concentration range of analytes of interest used to choose the appropriate analytical protocol.

CLASS A GLASSWARE - Defined by ASTM standards as glassware used in measurement with the smallest degree of uncertainty or tolerance associated with a measurement of volume.

CLOSING CONTINUING CALIBRATION VERIFICATION - Last analytical standard analyzed every 12 hours to verify the initial calibration accuracy of the system.

CONCENTRATION LEVEL (trace, low, or medium) - Characterization of sample fractions as trace concentration, low concentration, or medium concentration is made on the basis of the laboratory's preliminary screen, not on the basis of information entered on the Traffic Report/Chain-of-Custody (TR/COC) Record by the sampler.

CONTAMINATION - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION VERIFICATION (CCV) – A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards.

CONTINUOUS LIQUID-LIQUID EXTRACTION (CLLE) - 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.

CONTRACT COMPLIANCE SCREENING (CCS) - A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under EPA direction by the SMO Contractor.

CONTRACT LABORATORY PROGRAM (CLP) - Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of the EPA.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW), and supported by the analysis of standards.

CONTROL LIMITS – A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

DATE - The date format for all reporting forms is MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 2015, 2016, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) - Compound chosen to establish mass spectral instrument performance check for semivolatile analysis.

DEUTERATED MONITORING COMPOUNDS (DMCs) - Compounds added to every calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge-and-trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

DRY WEIGHT – The weight of a sample based on percent solids. The weight after drying in an oven.

EPA ASB ORGANIC CLP CONTRACTING OFFICER'S REPRESENTATIVE (ASB CLP COR) - The EPA ASB official who manages the Organic CLP Program.

EPA CONTRACTING OFFICER (CO) – The EPA official who has the authority to enter into, administer, terminate contracts, and/or make related determinations and findings.

EPA REGIONAL CLP CONTRACTING OFFICER'S REPRESENTATIVE (REGIONAL CLP COR) – The EPA official who monitors assigned CLP laboratories (either inside or outside of the Regional CLP COR's respective Region), responds to and identifies problems in laboratory operations, and participants in on-site laboratory audits.

EPA SAMPLE NUMBER - A unique identification number designated by the EPA for each sample. The EPA Sample Number appears on the Sample Traffic Report/Chain of Custody Record which documents information on that sample.

EXTRACTABLE - A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to Gas Chromatography. Extractables include semivolatile (SVOA), pesticide (PEST), and Aroclor (ARO) compounds.

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

FIELD BLANK - Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

FIELD QC - Any Quality Control (QC) samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, field duplicates, and field spikes.

FIELD SAMPLE - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

FORM - A hardcopy and/or electronic information/data entry sheet with locked preformatted structure that guides and/or controls user entry/input.

GAS CHROMATOGRAPH (GC) - The instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil), volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA, PEST, and ARO). In VOA and SVOA analysis, the analytes are detected by a Mass Spectrometer (MS). In Pesticide and Aroclor analysis, the analytes are detected by an Electron Capture Detector (ECD).

GAS CHROMATOGRAPH/ELECTRON CAPTURE DETECTOR - A Gas Chromatograph (GC) equipped with an Electron Capture Detector (ECD). This is one of the most sensitive gas chromatographic detectors for halogen-containing compounds such as organochlorine pesticides and polychlorinated biphenyls.

GAS CHROMATOGRAPH/MASS SPECTROMETER - A specialized form of Gas Chromatography (GC) used in conjunction with Mass Spectrometry (MS). GC/MS is considered the method of choice for the unequivocal identification of many volatile and semivolatile organic compounds.

GEL PERMEATION CHROMATOGRAPHY (GPC) - A size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

HOLDING TIME – Contractual holding time is the elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its extraction and analysis.

Holding time = (sample extraction date or analysis date - sample receipt date)

HYDROMATRIX™ - Diatomaceous earth-based material that is capable of adsorbing and retaining up to twice its weight of an aqueous media.

INDEPENDENT STANDARD – A Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the calibration.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions obtained from a source separate from that utilized to prepare the calibration standards, or a different lot than that used for the initial calibration (ICAL) standard. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.

INJECTION – Introduction of the analytical samples into the instrument excitation system to measure concentration of an analyte.

INSTRUMENT BLANK - A blank designed to determine the level of contamination associated with the analytical instruments.

INSUFFICIENT QUANTITY - When there is not enough volume (aqueous/water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, Percent Solids (%Solids), Matrix Spike and Matrix Spike Duplicate (MS/MSD), etc. Exhibit A – Summary of Requirements provides guidance for addressing this situation.

INTEGRATION SCAN RANGE - The scan number of the scan at the beginning of the area of integration to the scan number at the end of the area of integration.

INTEGRATION TIME RANGE - The Retention Time (RT) at the beginning of the area of integration to the RT at the end of the area of integration.

INTERFERENTS - Substances which affect the analysis for the element of interest.

INTERNAL STANDARDS - Compounds added to every standard, blank, Matrix Spike and Matrix Spike Duplicate (MS/MSD), sample (for volatiles), and sample extract aliquot (for semivolatiles) at a known concentration, prior to analysis. Instrument responses to internal standards are used as the basis for quantitation of the target compounds.

K-D – Kuderna-Danish concentrator; a device used to concentrate the analytes in a solvent.

LABORATORY - Synonymous with Contractor, as used herein.

LABORATORY CONTROL SAMPLE (LCS) - A reference matrix spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record. Also referred to as Validated Time of Sample Receipt (VTSR).

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

MATRIX EFFECT - In general, the effect of a particular matrix on the constituents under study. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may affect the purge and extraction efficiencies and consequently cause interference for the sample analyses.

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

MATRIX SPIKE DUPLICATE - A second aliquot of the same sample as the Matrix Spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - An aliquot of reagent water, silica sand, or corn oil that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The Method Blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

m/z - Mass to charge ratio; synonymous with "m/e".

OPENING CONTINUING CALIBRATION VERIFICATION - First analytical standard analyzed every 12 hours to verify the initial calibration of the system.

PERCENT DIFFERENCE (%D) - The difference between the two values divided by one of the values multiplied by 100.

PERCENT RECOVERY (%R)– The percentage of an analyte or element added to a sample that is recovered. It is the difference between the concentration detected in the spiked sample and that detected in the original (unspiked) sample, divided by the concentration added to the spiked sample multiplied by 100.

PERCENT SOLIDS (%S) – The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION MIXTURE (PEM) - A calibration solution of specific analytes used to evaluate both recovery and Percent Breakdown (%Breakdown) as a measure of performance.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition to the EPA; however, unknown to the Contractor that is provided to evaluate Contractor performance.

PREPARATION BLANK - An analyte-free sample to which all reagents are added in the same volume or proportions as used in sample processing. The preparation blank must be carried through the entire sample preparation and analytical procedures. It is used to assess contamination resulting from the analytical process.

PREPARATION LOG - An official record of the sample preparation.

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

PROFICIENCY TESTING (PT) AUDIT SAMPLE - A sample of known composition provided by the EPA for Contractor analysis. Used by the EPA to evaluate Contractor performance on a program-wide basis.

PURGE-AND-TRAP (DEVICE) - Analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

PURGEABLES - Volatile compounds.

QUALITY ASSURANCE TECHNICAL SUPPORT (QATS) LABORATORY - A Contractor-operated facility operated under the QATS contract, awarded and administered by the EPA.

RAW DATA - The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc.

REAGENT WATER - The purity of this water must be equivalent to ASTM Type II reagent water of Specification D1193-06, "Standard Specification for Reagent Water".

RECONSTRUCTED ION CHROMATOGRAM (RIC) - A mass spectral graphical representation of the separation achieved by a Gas Chromatograph (GC); a plot of total ion current versus Retention Time (RT).

RELATIVE PERCENT DIFFERENCE (RPD) - 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).

RELATIVE RESPONSE FACTOR (RRF) - The ratio of the response of a given compound to its corresponding internal standard. Response factors are determined using the area responses of both the primary and secondary exact m/z for each compound in each calibration standard.

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

REPORTED DATA - Reported data are processed from the raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements, such as significant figures and decimal precision.

RESOLUTION - Also termed Separation or Percent Resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESOLUTION CHECK MIXTURE - A solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

RESPONSE (Instrumental Response) - A measurement of the output of the Gas Chromatograph (GC) detector [Mass Spectrometer (MS), or Electron Capture Detector (ECD)] in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

RETENTION TIME (RT) - The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target analyte's retention time falling within the specified retention time window established for that analyte. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

ROUNDING RULES - If the figure following those to be retained is greater than or equal to 5, round up; otherwise, round down. As an example, 11.443 is rounded down to 11.4 and 11.455 is rounded up to 11.5. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures. See specific form instructions (Exhibit B – Reporting and Deliverables Requirements) for exceptions.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- All samples scheduled with the same level of deliverables.
- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory. Laboratories shall take all precautions to meet the 20 sample per SDG criteria.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by the EPA.

SDG NARRATIVE – Portion of the data package which includes laboratory, contract, Case, and Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete Sample Delivery Group (SDG) Narrative specifications are included in Exhibit B – Reporting and Deliverables Requirements.

SECONDARY QUANTITATION ION - Contract specified ion(s) to be used in quantitation of target analytes when interferences prevent the use of the primary quantitation ion.

SELECTED ION MONITORING (SIM) – A mode of Mass Spectrometry (MS) operation in which specific m/e ratios are monitored, as opposed to scanning the entire mass range.

SEMIVOLATILE COMPOUNDS - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral and Acid (BNA) compounds.

SENSITIVITY – The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

SOIL - Synonymous with soil/sediment as used herein.

STANDARD ANALYSIS - An analytical determination made with known quantities of target compounds; used to determine response factors.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

STORAGE BLANK - Reagent water (two 40.0 mL aliquots) stored with volatile samples in an SDG. It is analyzed after all samples have been analyzed in the SDG and is used to determine the level of contamination acquired during storage.

SULFUR BLANK - A modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur blank is required.

SUPPORTING DATA – Any data that substantiates the Reported Data (see definition above), including initial instrument measurements, instrument result calculations, standards concentrations, standard concentration calculations, sample preparation data (e.g., initial/final sample volume measurements, reagent quantities, etc.), and Method Detection Limits (MDLs). Supporting Data include standard preparation logs, sample preparation logs, instrument analysis logs, MDL and IEC studies, balance logs, pipette logs, percent solids logs, etc.

SURROGATES (Surrogate Standard) - For pesticides and Aroclors, compounds added to every blank, sample, Matrix Spike and Matrix Spike Duplicates (MS/MSDs), and standard. Surrogates are used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TARGET ANALYTE LIST - A list of analytes as designated by the Statement of Work (SOW) for analysis. See Exhibit C - Organic Target Analyte List and Contract Required Quantitation Limits.

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - Compounds detected in samples that are not target compounds, internal standards, Deuterated Monitoring Compounds (DMCs), or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of the nearest internal standard) are subjected to mass spectral library searches for tentative identification.

TIME - hh:mm:ss - When required to record time on any deliverable item, time shall be expressed as Military Time [i.e., a 24-hour clock (0000-2359)].

TRAFFIC REPORT/CHAIN OF CUSTODY RECORD (TR/COC) - An EPA sample identification form completed by the sampler, which accompanies the sample during shipment to the laboratory and is used to document sample identity, sample chain of custody, sample condition, and sample receipt by the laboratory.

TWELVE-HOUR TIME PERIOD - The 12-hour time period for Gas Chromatograph/Mass Spectrometer (GC/MS) system instrument performance check, standards calibration (initial, initial calibration verification, or continuing calibration), and method blank analysis begins at the moment of injection of the Decafluorotriphenylphosphine (DFTPP) or 4-Bromofluorobenzene (BFB) analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide and Aroclor analyses performed by Gas Chromatography/Electron Capture Detection (GC/ECD), the 12-hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after 12 hours have elapsed according to the system clock.

ULTRASONIC CELL DISRUPTOR (SONICATOR) - A device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given matrix.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor’s facility, as recorded on the shipper’s delivery receipt and sample Traffic Report/Chain of Custody Record.

VOLATILE COMPOUNDS - Compounds amenable to analysis by the purge-and-trap technique. Used synonymously with purgeable compounds.

WET WEIGHT - The weight of a sample aliquot including moisture (undried).

WIDE-BORE CAPILLARY COLUMN - A Gas Chromatographic column with an Internal Diameter (ID) that is greater than or equal to 0.53 mm. Columns with lesser diameters are classified as narrow bore capillary columns.

EXHIBIT H

FORMAT FOR ELECTRONIC DATA DELIVERABLES

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Table of Contents

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1.0 INTRODUCTION

The organic analytical service provides analytical data for use by the U.S. Environmental Protection Agency (EPA), in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). The electronic data deliverable (EDD) requirements in this section are designed to allow the EPA and other federal agencies or programs to rapidly assess the accuracy, completeness, and usefulness of the analytical results and the data. Depending on the stage chosen, the data user will receive results, support quality control (QC), and verification of the calculated results and quality measures.

2.0 FORMAT CHARACTERISTICS

2.1 This constitutes an implementation of the Staged Electronic Data Deliverable (SEDD) based on analytical results and other associated information required by the contract. Because this implementation is specific to the contract, not all data elements listed in the cross-program Document Type Definition (DTD) are required. This implementation is based on SEDD Specification 5.2 that can be found at:

https://www.epa.gov/clp/staged-electronic-data-deliverable-sedd

2.1.1 The SEDD deliverable consists of an eXtensible Markup Language (XML) file(s) compliant with the XML specification 1.0 of the World Wide Web Consortium (W3C). The deliverable must be well-formed based on the W3C XML specification and must be valid based on the DTD.

2.1.2 The Contractor shall create the deliverable using the UTF-8 (Unicode Transformation Format - 8 bit) character set.

2.1.3 The EDD SEDD stage delivery level (2a, 2b, or 3) must match the EPA requested/scheduled EDD SEDD level.

2.1.4 The initial line of the deliverable shall be: <?xml version="1.0" encoding="UTF-8"?>.

2.1.5 The second line of the deliverable shall be a DOCTYPE line that contains the filename of the DTD. The DOCTYPE line shall be <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_3_3.dtd">, <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_2b_3.dtd">, or <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_2a_2.dtd">, where "Header" denotes the name of the root element, and "SEDD_5-2_GENERAL_3_3.dtd" (for a Level 3 deliverable), "SEDD_5-2_GENERAL_2b_3.dtd" (for a Level 2b deliverable), or "SEDD_5-2_GENERAL_2a_2.dtd" (for a Level 2a deliverable) denotes the filename of the DTD.

2.1.6 The use of XML comment lines is permitted at any position in the file after the first two lines.

2.2 This implementation includes detailed specifications for the required format of the content of each data element for each analytical method. The content of each data element is specified as either literal (contained in quotes) which must appear exactly as shown (without quotes), or as a variable for which descriptions and formats are listed. Exhibit H, Section 3.0 describes the requirements for each data element.

2.2.1 For this implementation, numeric data elements may contain numeric digits, a decimal place, and a leading minus sign. Values without a leading minus sign are assumed to be positive. Values must be reported to the specified precision or significance.

2.2.2 The values reported by the Contractor are used for data assessment. No raw data values in the SEDD files shall be rounded. The Contractor shall not use rounded intermediate values in calculating the final result, and no rounding shall be performed until reaching the final result.

2.2.3 The completeness of analytical data provided in the EDD will be verified against the analytical data requested on the Traffic Report/Chain of Custody (TR/COC) Record. The Laboratory Code, Case Number, Contract Number, Sample Delivery Group (SDG) Number, Modified Analysis (MA) Number (if applicable), sample number, and analytical method shall be identical in the EDD and the TR/COC Record and the SDG coversheet submitted by the Contractor for the SDG.

2.2.4 The following variables shall be present where required and correct: EDD Implementation Identifier (ID); Lab ID; Lab Receipt Date; Analysis Date and Time; Collected Date; Matrix ID; Client Method ID; Client Method Type; QC Type; Instrument ID; Relative Response Factor (RRF) or Calibration Factor (CF); mean RRF or mean CF; Run Batch (level 2b and 3 only); Analysis Batch (level 2b and 3 only); Analysis Group ID (level 2b and 3 only); Client Analysis ID; Client Analyte ID; Preparation Batch; Percent Recovery (%R); Relative Percent Difference (RPD); Percent Difference (%D); and Percent Relative Standard Deviation (%RSD).

3.0 DATA ELEMENTS

3.1 The SEDD consists of data elements arranged hierarchically by data nodes (parent elements). Figures 1, 2, and 3 depict the data node hierarchy. Each data element consists of a start tag, content, and an end tag. An element may contain other elements (child elements).

NOTE: There shall be no more than one occurrence of each child element within a node, unless the child element also behaves as a parent element. For example, in each SamplePlusMethod node, there may be only one occurrence of the element ClientSampleID, but there may be more than one occurrence of the element Analysis.

The tags, nodes, and hierarchy are specified in the DTD against which the deliverable will be validated (see Exhibit H, Section 6.0). The frequency requirements for each of the data nodes applicable to this implementation are described below.

3.1.1 Header Node (Required for All Deliverable Levels)

One Header node must be reported for each analytical method.

3.1.2 SamplePlusMethod Node (Required for All Deliverable Levels)

Each Header node must contain one SamplePlusMethod node for each field sample, field blank (including rinse, equipment, and trip blanks), Performance Evaluation (PE) sample, Proficiency Testing (PT) audit sample, Matrix Spike (MS) sample, Matrix Spike Duplicate (MSD) sample, Method Blank (MB), Leachate Extraction Blank (LEB), Instrument Blank (IB), Storage Blank (SB) (Volatiles only), Cleanup Blank (CB) (Pesticides and Aroclors only), Laboratory Control Sample (LCS), and Non-Client Sample (NCS).

3.1.3 ReportedResult Node (Required for All Deliverables Levels)

Each SamplePlusMethod node must contain one and only one ReportedResult node for each target analyte. For Volatiles and Semivolatiles, each SamplePlusMethod node must also contain a ReportedResult node for each Tentatively Identified Compound (TIC).

3.1.4 ContactInformation Node (Required for All Deliverable Levels)

Each Header node must contain one ContactInformation node.

3.1.5 InstrumentQC Node (Required for Levels 2b and 3 Deliverables Only)

Each Header node must contain one InstrumentQC node for each instrument performance check, initial calibration sequence, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Florisil Cartridge Check, and Gel Permeation Chromatography (GPC) Calibration Check.

NOTE: Tunes may be reported as separate InstrumentQC nodes or may be included in InstrumentQC nodes for initial calibration or CCV. This will depend on whether the tune is analyzed as a separate injection or is combined with a calibration standard.

3.1.6 AnalysisGroup Node (Required for Levels 2b and 3 Deliverables Only)

Each initial calibration InstrumentQC node for multi-point calibration must contain one AnalysisGroup node containing summary data for the initial calibration. Each AnalysisGroup node must contain one Analyte node for each target analyte, Deuterated Monitoring Compound (DMC), and surrogate.

3.1.7 Analysis Node (Required for All Deliverable Levels)

Each SamplePlusMethod must contain at least one Analysis node for Gas Chromatograph/Mass Spectrometer (GC/MS) methods or must contain at least two Analysis nodes for GC methods with confirmation (one for each column). Separate Analysis nodes are required for each dilution, re-extraction, or reanalysis. Any reanalysis must be preceded by an initial analysis. Any analysis reported as a dilution must also have a less-diluted analysis reported as initial. The initial analysis does not have to precede the diluted analysis.

Each Instrument QC node (other than Initial Calibration) must contain one Analysis node for GC/MS methods or must contain at least two Analysis nodes for GC methods with confirmation (one for each column).

3.1.8 Analyte Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain one Analyte node for each target analyte, TIC, DMC, surrogate, and internal standard. Each Analysis node under an InstrumentQC node must contain one Analyte node for each target analyte, DMC, surrogate, and internal standard. Each Analysis node under an InstrumentQC node for tune must contain one Analyte node for each tune analyte. Each AnalysisGroup node must contain one Analyte node for each target analyte, DMC, and surrogate.

3.1.9 PreparationPlusCleanup Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain at least one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation", and one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Cleanup" for each applicable cleanup technique performed. Each Analysis node under an InstrumentQC node with a QCType equal to "Florisil_Cartridge_Check" or "GPC_Calibration_Check" must contain one PreparationPlusCleanup node. No more than one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation" shall be present for each analysis.

3.1.10 Peak Node (Required for Levels 2b and 3 Deliverables Only)

Each Analyte node must contain at least one Peak node, and a minimum of 3 Peak nodes for Toxaphene and Aroclors. For Level 2b, only the Analyte nodes under InstrumentQC must contain a Peak node. Within a RunBatch, a peak must be consistently identified.

3.1.11 PeakComparison Node (Required for Levels 2b and 3 Deliverables Only)

For GC/MS, each Peak node must contain a PeakComparison node for each applicable internal standard.

3.1.12 Characteristic Node (Required for All Deliverable Levels)

Each SamplePlusMethod, PreparationPlusCleanup, and Handling node may contain one or more Characteristic nodes, one for each sample characteristic that must be reported for a sample at time of receipt, after preparation, or after handling.

3.1.13 Handling Node (required for Level 3 Deliverables only)

Each SamplePlusMethod node shall contain one or more Handling nodes when Toxicity Characteristic Leaching Procedure (TCLP) extraction, Synthetic Precipitation Leaching Procedure (SPLP) extraction, or decanting has been performed.

3.2 Detailed instructions for the content of each data element are provided in Section 7.0, Tables 1 through 6. The following is an explanation of the data fields contained in each table.

3.2.1 Node and Data Elements

This field reports each node in bold text, followed by its data elements. If an entire node is not required, then none of its data elements are listed.

3.2.2 Applicability

This field reports the samples, blanks, and standards for which each node and data element is required. An "X" in a column indicates that the node or element is required. Sample refers to field samples, field blanks, and PE samples unless otherwise noted. Abbreviations used in this field are defined in Section 7.0, Table 7 - Abbreviations.

3.2.3 Instructions

This field describes the required format and content of each data element. The content of each data element is specified as either literal (contained in quotes), or as a variable for which description and format is listed. Abbreviations used in this field are defined in Section 7.0, Table 7 - Abbreviations.

Figure 1: Data Node Hierarchy for
Level 2a Deliverable

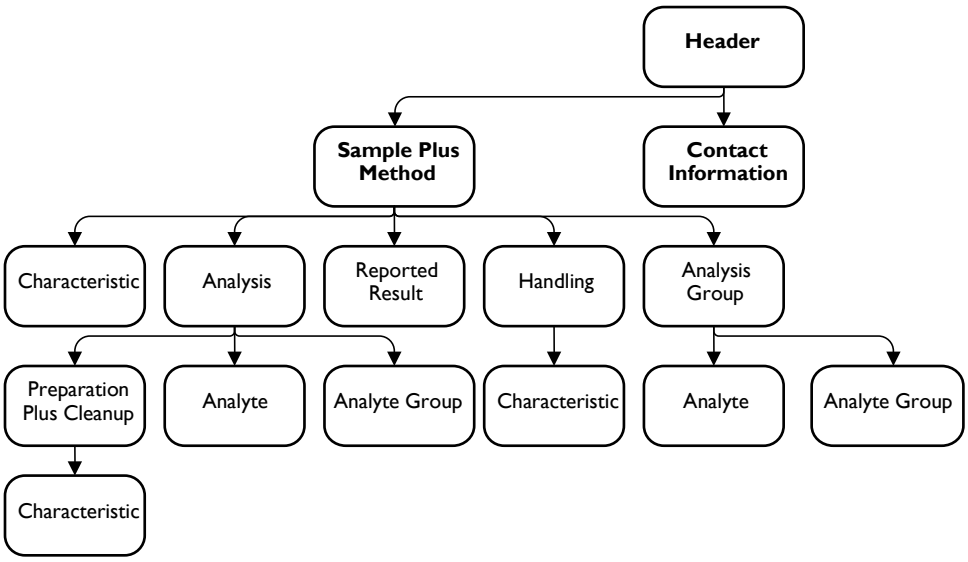


Figure 2: Data Node Hierarchy for
Level 2b Deliverable

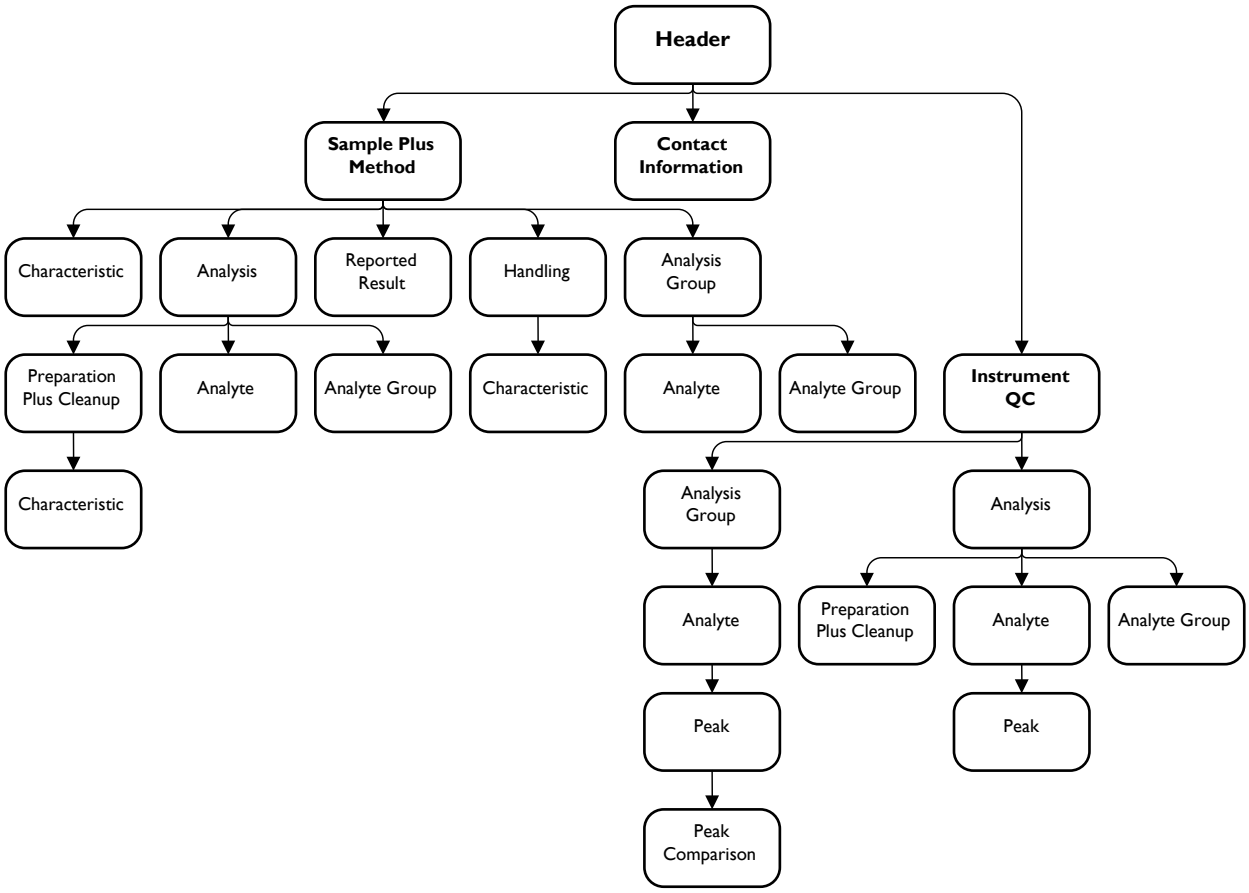
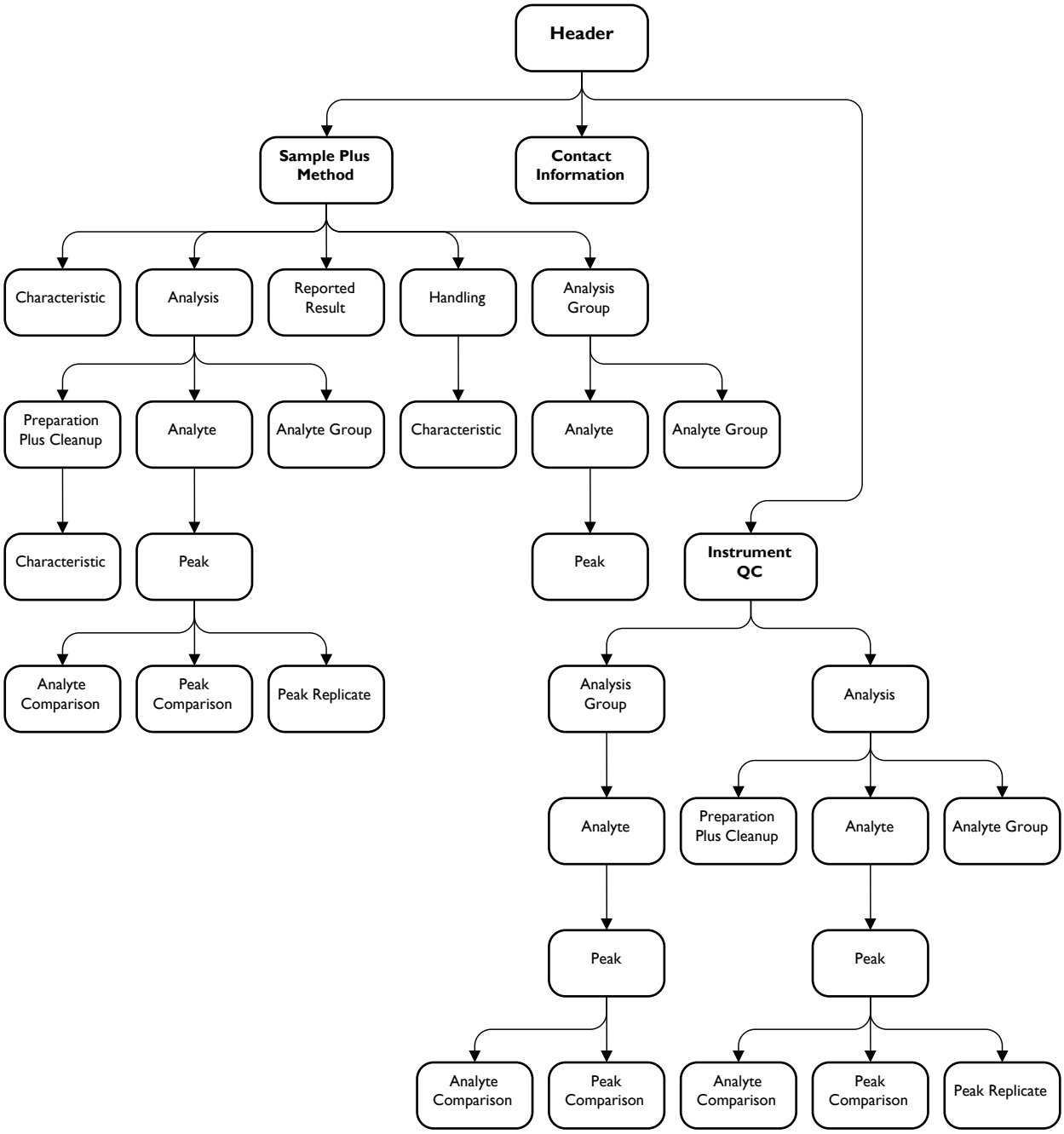


Figure 3: Data Node Hierarchy for
Level 3 Deliverable



4.0 BATCHES

4.1 This implementation requires the use of the following SEDD batches from the SEDD Specification: "LabReportingBatch"; "RunBatch"; "AnalysisBatch"; "PreparationBatch"; "HandlingBatch"; and "StorageBatch".

4.1.1 The "LabReportingBatch" links all samples reported in the same SDG. Report the SDG Number.

4.1.2 The "RunBatch" links all analyses performed under the same initial calibration. All analyses performed under an initial calibration must have the same content for the "RunBatch" element as the initial calibration from which their results are calculated.

4.1.3 The "AnalysisBatch" and "AnalysisBatchEnd" link all analyses performed within the same analytical sequence. All analyses performed within the same analytical sequence must have the same content for the "AnalysisBatch" element as the tune or standard(s) that began the analytical sequence, and the same content for the "AnalysisBatchEnd" as the standard(s) that ends the analytical sequence.

4.1.4 The "PreparationBatch" links all samples of the same matrix prepared at the same time by the same preparation method. All samples analyzed, including Method Blanks, MS/MSDs, and LCSs, that are prepared together must have the same content for the "PreparationBatch" element.

4.1.5 The "HandlingBatch" links all samples subjected to TCLP or SPLP extraction or decanting at the same time by the same method. All samples extracted, including the LEB, that are extracted together must have the same content for the "HandlingBatch" element.

4.1.6 The "StorageBatch" links all samples stored together with a storage blank. All samples that are stored together must have the same content for the "StorageBatch" element as the storage blank sample.

4.1.7 The "CleanupBatch" links all samples processed by the same cleanup method. All samples analyzed, including method blanks and LCS, that are cleaned up together must have the same content for the "CleanupBatch" element.

5.0 DELIVERABLE

5.1 Each analytical method in an SDG shall be submitted as a separate file. The Contractor may choose to deliver their file as a ZIP of an XML file. For reporting requirements, the analytical methods are: "VOA_Trace"; "VOA_Low_Med"; "SVOA"; "SVOA_SIM"; "Pest"; and "Aroclor". For example, if Selected Ion Monitoring (SIM) is requested for semivolatile organic analytes (SVOA), then 2 separate files must be submitted and labeled as "SVOA" and "SVOA_SIM". All analytical methods within an SDG shall be submitted at the same time (i.e., the file for the second analytical method in an SDG shall be submitted in a single file upload with the first analytical method).

5.2 The Contractor will utilize the Electronic Data Exchange and Evaluation System (EXES) at https://epasmoweb.fedcsc.com to electronically submit their EDD to the Sample Management Office (SMO). The EPA may approve alternative electronic means of file delivery. Written permission must be obtained from the EPA Analytical Services Branch (ASB) prior to the use of any alternative means.

5.3 The Contractor must follow the delivery instructions in Exhibit B – Reporting and Deliverables Requirements, of this Statement of Work (SOW), and deliver their hardcopy and EDD and Portable Document Format (PDF) of the Complete SDG File (CSF) to SMO concurrently. If one of these items is delivered on a later date, the Data Receipt Date (DRD) for the SDG will be the later of the two dates.

5.4 Information in the electronic deliverable must correspond to information submitted in the hardcopy raw data package and on QC summary forms. If information in the raw data or on the forms is changed, the information in the electronic deliverable shall be changed accordingly. An electronic deliverable containing the changed information for the SDG shall be resubmitted along with the hardcopy at no additional cost to the EPA.

5.5 The format for the file name shall be Case number_SDG number_contract number_submission number_DTD used_Method. For example, the first submission of the Trace VOA Analytical Method from SDG number ABC12, Case number 12345, contract EP-W00-000 would be named 12345_ABC12_EP-W-00000_1_SEDD_5-2_GENERAL_3_3_VOA_Trace.zip.

6.0 DOCUMENT TYPE DEFINITION

6.1 Introduction

The deliverable will be validated against DTD SEDD_5-2_GENERAL_3_3, DTD SEDD_5-2_GENERAL_2b_3 or DTD SEDD_5-2_GENERAL_2a_2. The deliverable must not contain any tags not included in the DTD and must conform to the hierarchical structure modeled in the DTD.

6.2 General Stage 3 DTD

```
<?xml version="1.0" encoding="UTF-8"?>
<!-- SEDD_5-2_GENERAL_3_3.dtd 10/22/2009 -->
<!-- Acronym Description -->
<!-- Coeff - Coefficient -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!-- RRF   - Relative Response Factor -->
<!-- RSD   - Relative Standard Deviation -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod|
                InstrumentQC
                        )*>
<!ELEMENT Analysis (
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                AliquotAmountUnits|
                AnalysisBatch|
                AnalysisBatchEnd|
                AnalysisDuration|
```

AnalysisDurationUnits|
AnalysisGroupID|
AnalysisType|
Analyst|
AnalyzedAmount|
AnalyzedAmountUnits|
AnalyzedDate|
BackgroundCorrection|
BackgroundRawData|
BackgroundType|
BottleID|
ClientAnalysisID|
ClientMethodCode|
ClientMethodID|
ClientMethodModificationDescription|
ClientMethodModificationID|
ClientMethodName|
ClientMethodSource|
ClientMethodVersion|
Column|
ColumnInternalDiameter|
ColumnInternalDiameterUnits|
ColumnLength|
ColumnLengthUnits|
Comment|
ConfirmationAnalysisID|
Counts|
CountsUncertainty|
CountsUncertaintyConfidenceLevel|
CountsUncertaintyDetermination|
CountsUncertaintyIntervalType|
CountsUncertaintyLimitHigh|
CountsUncertaintyLimitLow|
CountsUncertaintyType|
CountsUnits|
DetectorID|
DetectorType|
DilutionFactor|
Efficiency|
HeatedPurge|
Inclusion|
InjectionVolume|
InjectionVolumeUnits|
InstrumentID|
InterelementCorrection|
LabAnalysisID|
LabFileID|
LabID|
LabMethodID|
LabMethodName|
LabName|
MethodCode|
MethodID|
MethodModificationDescription|
MethodModificationID|
MethodName|
MethodSource|
MethodVersion|
OriginalLabAnalysisID|
PreparationBatch|

```
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                RunBatch|
                SampleAmount|
                SampleAmountUnits|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT AnalysisGroup (
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                AnalysisType|
                Comment|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT Analyte (
                AmountAdded|
                AmountAddedUnits|
                AmountAddedLocation|
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CalibrationBasis|
                CalibrationFactor|
                CalibrationFactorUnits|
                CalibrationType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
                Comment|
                CorrelationCoeff|
                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
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                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectionLimit|
```

DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
Efficiency|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType|
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
Inclusion|
IntermediateResult|
IntermediateResultLimitHigh|
IntermediateResultLimitLow|
IntermediateResultLimitType|
IntermediateResultUnits|
LabAnalyteID|
LabQualifiers|
LotNumber|
Mass|
MassLimitHigh|
MassLimitLow|
MassLimitType|
MassUnits|
MeanCalibrationFactor|
MeanCalibrationFactorUnits|
MeanRRF|
MeanRRFLimitLow|
MeanRRFLimitType|
PeakID|
PercentBreakdown|
PercentBreakdownLimitHigh|
PercentBreakdownLimitType|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentMatch|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
PercentRSD|
PercentRSDLimitHigh|
PercentRSDLimitLow|
PercentRSDLimitType|
QuantitationBasis|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Response|
ResponseLimitHigh|

```
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                ResponseLimitType|
                ResponseUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
                ResultUncertaintyType|
                ResultUncertaintyUnits|
                ResultUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType|
                RRF|
                RRFLimitLow|
                RRFLimitType|
                StandardConcentration|
                StandardConcentrationUnits|
                StandardDeviation|
                StandardDeviationUnits|
                StandardFinalAmount|
                StandardFinalAmountUnits|
                StandardID|
                StandardSource|
                TailingFactor|
                TailingFactorLimitHigh|
                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                Peak
                        )*>
<!ELEMENT AnalyteComparison (
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                ClientAnalyteName|
                Comment|
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                LabAnalyteID
                        )*>
<!ELEMENT Characteristic (
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                CharacteristicValue|
                CharacteristicUnits|
                Comment
                        )*>
<!ELEMENT AnalyteGroup (
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
```

```
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                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                        )*>
<!ELEMENT ContactInformation (
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                LabAddress2|
                LabCity|
                LabCountry|
                LabID|
                LabName|
                LabPointOfContact|
                LabPointOfContactElectronicAddress|
                LabPointOfContactTitle|
                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
                        )*>
<!ELEMENT Handling (
                Analyst|
                BottleID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                HandledDate|
                HandlingBatch|
                HandlingType|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                ProcedureID|
                ProcedureName|
                SampleAmount|
                SampleAmountUnits|
                Characteristic
```

```
                        )*>
<!ELEMENT InstrumentQC (
                ClientInstrumentQCType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                LabID|
                LabInstrumentQCID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                QCLinkage|
                QCType|
                AnalysisGroup|
                Analysis
                        )*>
<!ELEMENT Peak (
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                CalibrationFactorUnits|
                CalibrationType|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
                Comment|
                CorrelationCoeff|
                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
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                Efficiency|
                Inclusion|
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                IntermediateResultLimitLow|
                IntermediateResultLimitType|
                IntermediateResultUnits|
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                ManualIntegration|
                Mass|
                MassLimitHigh|
                MassLimitLow|
                MassLimitType|
```

```
MassUnits|
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MeanCalibrationFactorUnits|
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MeanRetentionTimeLimitType|
MeanRetentionTimeUnits|
MeanRRF|
MeanRRFLimitLow|
MeanRRFLimitType|
PeakID|
PeakRatio|
PeakRatioLimitHigh|
PeakRatioLimitLow|
PeakRatioLimitType|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRatio|
PercentRatioLimitHigh|
PercentRatioLimitLow|
PercentRatioLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
PercentRSD|
PercentRSDLimitHigh|
PercentRSDLimitLow|
PercentRSDLimitType|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Resolution|
ResolutionLimitHigh|
ResolutionLimitLow|
ResolutionLimitType|
ResolutionType|
ResolutionUnits|
Response|
ResponseLimitHigh|
ResponseLimitLow|
ResponseLimitType|
ResponseType|
ResponseUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
ResultLimitType|
ResultType|
ResultUncertainty|
ResultUnits|
RetentionTime|
RetentionTimeLimitHigh|
```

```
                RetentionTimeLimitLow|
                RetentionTimeLimitType|
                RetentionTimeUnits|
                RRF|
                RRFLimitLow|
                RRFLimitType|
                StandardDeviation|
                StandardDeviationUnits|
                TailingFactor|
                TailingFactorLimitHigh|
                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                AnalyteComparison|
                PeakComparison|
                PeakReplicate
                         )*>
<!ELEMENT PeakComparison (
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                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                PeakID|
                PeakRatio|
                PeakRatioLimitHigh|
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                PeakRatioLimitType|
                PercentRatio|
                PercentRatioLimitHigh|
                PercentRatioLimitLow|
                PercentRatioLimitType
                         )*>
<!ELEMENT PeakReplicate (
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                IntermediateResultLimitHigh|
                IntermediateResultLimitLow|
                IntermediateResultLimitType|
                IntermediateResultUnits|
                Mass|
                MassLimitHigh|
                MassLimitLow|
                MassLimitType|
                MassUnits|
                PeakReplicateID|
                Resolution|
                ResolutionLimitHigh|
                ResolutionLimitLow|
                ResolutionLimitType|
                ResolutionType|
                ResolutionUnits|
                Response|
                ResponseLimitHigh|
                ResponseLimitLow|
                ResponseLimitType|
                ResponseType|
```

```
                ResponseUnits
                        )*>
<!ELEMENT PreparationPlusCleanup (
                AliquotAmount|
                AliquotAmountUnits|
                Analyst|
                BottleID|
                CleanedUpDate|
                CleanupBatch|
                CleanupType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                Efficiency|
                FinalAmount|
                FinalAmountUnits|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LotNumber|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                PreparationPlusCleanupType|
                PreparationType|
                PreparedDate|
                ProcedureID|
                ProcedureName|
                SampleAmount|
                SampleAmountUnits|
                Solvent|
                Characteristic
                        )*>
<!ELEMENT ReportedResult (
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                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                ClientDetectionLimit|
                ClientDetectionLimitUnits|
                ClientQuantitationLimit|
                ClientQuantitationLimitUnits|
```

```
                Comment|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
                DifferenceErrorRatio|
                ExpectedResult|
                ExpectedResultUncertainty|
                ExpectedResultUncertaintyConfidenceLevel|
                ExpectedResultUncertaintyDetermination|
                ExpectedResultUncertaintyIntervalType|
                ExpectedResultUncertaintyLimitHigh|
                ExpectedResultUncertaintyLimitLow|
                ExpectedResultUncertaintyType|
                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
                LabAnalysisID|
                LabAnalyteID|
                LabQualifiers|
                LabResultStatus|
                PeakID|
                PercentDifference|
                PercentDifferenceLimitHigh|
                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
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                PercentRecoveryLimitHigh|
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                ReportingLimitUnits|
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                ResultLimitLow|
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                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
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                ResultUncertaintyType|
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ClientMethodType|
ClientMethodVersion|
ClientName|
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CollectedEndDate|
Comment|
Composite|
CoolerID|
CustodyID|
EquipmentBatch|
Filtered|
LabContract|
LabContractModificationID|
LabContractModificationDescription|
LabID|
LabMethodID|
LabMethodName|
LabName|
LabReceiptDate|
LabReportingBatch|
LabSampleID|
LocationID|
LocationName|
MatrixID|
MatrixMedium|
MethodBatch|
MethodCategory|
MethodCode|
MethodID|
MethodLevel|
MethodModificationDescription|
MethodModificationID|
MethodName|
MethodSource|
MethodType|
MethodVersion|
OriginalClientSampleID|
OriginalLabSampleID|
PhaseAnalyzed|
Preservative|
ProjectID|
ProjectName|
QCCategory|
QCLinkage|
QCType|
Quarantine|
SamplingBatch|
ShippingBatch|
SiteID|
SiteName|
StorageBatch|
Analysis|

```
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                AnalysisGroup|
                Characteristic
                          )*>
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```
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6.3 General Stage 2b DTD

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<!—SEDD_5-2_GENERAL_2b_3.dtd 10/22/2009 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- Coeff - Coefficient -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!-- RRF   - Relative Response Factor -->
<!-- RSD   - Relative Standard Deviation -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
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                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod|
                InstrumentQC
                        )*>
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                AnalysisDuration|
                AnalysisDurationUnits|
                AnalysisGroupID|
                AnalysisType|
                Analyst|
                AnalyzedAmount|
                AnalyzedAmountUnits|
                AnalyzedDate|
                ClientAnalysisID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
```

```
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                ClientMethodSource|
                ClientMethodVersion|
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                ColumnInternalDiameterUnits|
                ColumnLength|
                ColumnLengthUnits|
                Comment|
                ConfirmationAnalysisID|
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                CountsUncertainty|
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                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
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                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
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                DetectorType|
                DilutionFactor|
                Efficiency|
                HeatedPurge|
                Inclusion|
                InjectionVolume|
                InjectionVolumeUnits|
                InstrumentID|
                LabAnalysisID|
                LabFileID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                RunBatch|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT AnalysisGroup (
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                AnalysisType|
                Comment|
```

```
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                AnalyteGroup
                        )*>
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                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
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                CalibrationBasis|
                CalibrationFactor|
                CalibrationFactorUnits|
                CalibrationType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
                Comment|
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                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
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                CountsUncertaintyDetermination|
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                CountsUncertaintyType|
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                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
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                MeanCalibrationFactorUnits|
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                MeanRRFLimitLow|
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                PercentBreakdownLimitType|
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                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
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                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
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                PercentRSDLimitLow|
                PercentRSDLimitType|
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                QuantitationLimit|
                QuantitationLimitType|
                QuantitationLimitUnits|
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                ReportingLimitUnits|
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                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
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                ResultUncertaintyType|
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                ResultUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType|
                RRF|
                RRFLimitLow|
                RRFLimitType|
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                TailingFactorLimitType|
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                WavelengthUnits|
                WeightingFactor|
                Peak
                         )*>
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                AnalyteType|
                CASRegistryNumber|
```

```
                ClientAnalyteID|
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                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                        )*>
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                        )*>
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                LabCity|
                LabCountry|
                LabID|
                LabName|
                LabPointOfContact|
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                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
                        )*>
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                HandlingType|
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                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
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                MethodSource|
                MethodVersion|
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                ProcedureName|
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```
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                        )*>
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                LabMethodID|
                LabMethodName|
                LabName|
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                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                QCLinkage|
                QCType|
                AnalysisGroup|
                Analysis
                        )*>
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                Coeffa2|
                Coeffa3|
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                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
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                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
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                Efficiency|
                Inclusion|
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                MassLimitLow|
                MassLimitType|
                MassUnits|
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                MeanCalibrationFactorUnits|
                MeanRetentionTime|
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                MeanRetentionTimeLimitLow|
                MeanRetentionTimeLimitType|
                MeanRetentionTimeUnits|
```

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                PercentDifferenceLimitType|
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                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                PercentRSD|
                PercentRSDLimitHigh|
                PercentRSDLimitLow|
                PercentRSDLimitType|
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                ResolutionLimitLow|
                ResolutionLimitType|
                ResolutionType|
                ResolutionUnits|
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                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUnits|
                RRF|
                RRFLimitLow|
                RRFLimitType|
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                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                PeakComparison
                        )*>
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                PercentRatioLimitLow|
                PercentRatioLimitType
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                CleanedUpDate|
                CleanupBatch|
                CleanupType|
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                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
```

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                FinalAmountUnits|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LotNumber|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                PreparationPlusCleanupType|
                PreparationType|
                PreparedDate|
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                ProcedureName|
                Solvent|
                Characteristic
                        )*>
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                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                ClientDetectionLimit|
                ClientDetectionLimitUnits|
                ClientQuantitationLimit|
                ClientQuantitationLimitUnits|
                Comment|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
                DifferenceErrorRatio|
                ExpectedResult|
                ExpectedResultUncertainty|
                ExpectedResultUncertaintyConfidenceLevel|
                ExpectedResultUncertaintyDetermination|
                ExpectedResultUncertaintyIntervalType|
                ExpectedResultUncertaintyLimitHigh|
                ExpectedResultUncertaintyLimitLow|
                ExpectedResultUncertaintyType
                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
                LabAnalysisID|
                LabAnalyteID|
                LabQualifiers|
                LabResultStatus|
```

```
                PeakID|
                PercentDifference|
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                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
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                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                QuantitationLimit|
                QuantitationLimitType|
                QuantitationLimitUnits|
                ReportingLimit|
                ReportingLimitType|
                ReportingLimitUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
                ResultUncertaintyType|
                ResultUncertaintyUnits|
                ResultUnits|
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                RetentionTimeUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType
                          )*>
<!ELEMENT SamplePlusMethod (
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                ClientMethodCode|
                ClientMethodID|
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                ClientMethodName|
                ClientMethodSource|
                ClientMethodType|
                ClientMethodVersion|
                ClientName|
                ClientSampleID|
                CollectedDate|
                CollectedEndDate|
                Comment|
                Composite|
                CoolerID|
                CustodyID|
                EquipmentBatch|
                Filtered|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
```

```
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                LabMethodID|
                LabMethodName|
                LabName|
                LabReceiptDate|
                LabReportingBatch|
                LabSampleID|
                LocationID|
                LocationName|
                MatrixID|
                MatrixMedium|
                MethodBatch|
                MethodCategory|
                MethodCode|
                MethodID|
                MethodLevel|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodType|
                MethodVersion|
                OriginalClientSampleID|
                OriginalLabSampleID|
                PhaseAnalyzed|
                Preservative|
                ProjectID|
                ProjectName|
                QCCategory|
                QCLinkage|
                QCType|
                Quarantine|
                SamplingBatch|
                ShippingBatch|
                SiteID|
                SiteName|
                StorageBatch|
                Analysis|
                Characteristic|
                ReportedResult|
                Handling|
                AnalysisGroup
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```

```
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```
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<!ELEMENT ResolutionType (#PCDATA)>
<!ELEMENT ResolutionUnits (#PCDATA)>
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<!ELEMENT ResultUncertaintyDetermination (#PCDATA)>
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<!ELEMENT SamplingBatch (#PCDATA)>
```

```
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<!ELEMENT SiteID (#PCDATA)>
<!ELEMENT SiteName (#PCDATA)>
<!ELEMENT Solvent (#PCDATA)>
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<!ELEMENT WavelengthUnits (#PCDATA)>
<!ELEMENT WeightingFactor (#PCDATA)>
<!ELEMENT Yield (#PCDATA)>
```

6.4 General Stage 2a DTD

```
<?xml version="1.0" encoding="UTF-8"?>
<!—SEDD_5-2_GENERAL_2a_2.dtd 07/21/2008 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod
                        )*>
<!ELEMENT Analysis (
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                AliquotAmountUnits|
                AnalysisDuration|
                AnalysisDurationUnits|
                AnalysisGroupID|
                AnalysisType|
                Analyst|
                AnalyzedAmount|
                AnalyzedAmountUnits|
                AnalyzedDate|
                ClientAnalysisID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Column|
```

```
                ColumnInternalDiameter|
                ColumnInternalDiameterUnits|
                ColumnLength|
                ColumnLengthUnits|
                Comment|
                ConfirmationAnalysisID|
                Counts|
                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectorID|
                DetectorType|
                DilutionFactor|
                Efficiency|
                HeatedPurge|
                Inclusion|
                InjectionVolume|
                InjectionVolumeUnits|
                InstrumentID|
                LabAnalysisID|
                LabFileID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT AnalysisGroup (
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                AnalysisType|
                Comment|
                Analyte|
                AnalyteGroup
                        )*>
```

```
<!ELEMENT Analyte (
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                Counts|
                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
                DifferenceErrorRatio|
                Efficiency|
                ExpectedResult|
                ExpectedResultUncertainty|
                ExpectedResultUncertaintyConfidenceLevel|
                ExpectedResultUncertaintyDetermination|
                ExpectedResultUncertaintyIntervalType|
                ExpectedResultUncertaintyLimitHigh|
                ExpectedResultUncertaintyLimitLow|
                ExpectedResultUncertaintyType|
                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
                Inclusion|
                LabAnalyteID|
                LabQualifiers|
                LotNumber|
                PeakID|
                PercentRecovery|
                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                QuantitationLimit|
                QuantitationLimitType|
                QuantitationLimitUnits|
                ReportingLimit|
                ReportingLimitType|
                ReportingLimitUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
```

```
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                ResultUnits|
                StandardSource|
                Wavelength|
                WavelengthUnits
                        )*>
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                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                        )*>
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                CharacteristicValue|
                CharacteristicUnits|
                Comment
                        )*>
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                LabCity|
                LabCountry|
                LabID|
                LabName|
                LabPointOfContact|
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                LabPointOfContactTitle|
                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
                        )*>
<!ELEMENT Handling (
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                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                HandledDate|
                HandlingBatch|
                HandlingType|
                InitialAmount|
```

```
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                ProcedureID|
                ProcedureName|
                SampleAmount|
                SampleAmountUnits|
                Characteristic
                        )*>
<!ELEMENT PreparationPlusCleanup (
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                AliquotAmountUnits|
                Analyst|
                CleanedUpDate|
                CleanupBatch|
                CleanupType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
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                FinalAmountUnits|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LotNumber|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                PreparationPlusCleanupType|
                PreparationType|
                PreparedDate|
                ProcedureID|
                ProcedureName|
                Solvent|
                Characteristic
                        )*>
```

```
<!ELEMENT ReportedResult (
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                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                ClientDetectionLimit|
                ClientDetectionLimitUnits|
                ClientQuantitationLimit|
                ClientQuantitationLimitUnits|
                Comment|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
                DifferenceErrorRatio|
                ExpectedResult|
                ExpectedResultUncertainty|
                ExpectedResultUncertaintyConfidenceLevel|
                ExpectedResultUncertaintyDetermination|
                ExpectedResultUncertaintyIntervalType|
                ExpectedResultUncertaintyLimitHigh|
                ExpectedResultUncertaintyLimitLow|
                ExpectedResultUncertaintyType|
                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
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                LabAnalyteID|
                LabQualifiers|
                LabResultStatus|
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                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
                PercentRecovery|
                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
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                QuantitationLimitUnits|
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                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
```

```
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                RPDLimitType|
                RPDType
                        )*>
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                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodType|
                ClientMethodVersion|
                ClientName|
                ClientSampleID|
                CollectedDate|
                CollectedEndDate|
                Comment|
                Composite|
                CoolerID|
                CustodyID|
                EquipmentBatch|
                Filtered|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LabReceiptDate|
                LabReportingBatch|
                LabSampleID|
                LocationID|
                LocationName|
                MatrixID|
                MatrixMedium|
                MethodBatch|
                MethodCategory|
                MethodCode|
                MethodID|
                MethodLevel|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodType|
                MethodVersion|
                OriginalClientSampleID|
                OriginalLabSampleID|
                PhaseAnalyzed|
```

```
                Preservative|
                ProjectID|
                ProjectName|
                QCCategory|
                QCLinkage|
                QCType|
                Quarantine|
                SamplingBatch|
                ShippingBatch|
                SiteID|
                SiteName|
                StorageBatch|
                Analysis|
                Characteristic|
                ReportedResult|
                Handling|
                AnalysisGroup
                          )*>
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<!ELEMENT ProjectName (#PCDATA)>
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<!ELEMENT QCType (#PCDATA)>
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```

7.0 DATA ELEMENT INSTRUCTION TABLES

Column abbreviations: Matrix Spike (MS), Matrix Spike Duplicate (MSD), Method Blank (MB), Leachate Extraction Blank (LEB), Storage Blank (SB), Instrument Blank (IB), Non-Client Sample (NCS), Cleanup Blank (CB), Laboratory Control Sample (LCS), Instrument Performance Check (IPC), Initial Calibration (ICAL), Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Florisil Cartridge Check (FLO), and Gel Permeation Chromatography Calibration Verification (GPC).

7.1 Stage 3

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Header	X	X	X	X	
ClientID	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName					Not required.
Comment					Not required.
DateFormat	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabDataPackageID	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	Report "VOA_Trace", "VOA_Low_Med", "SVOA", or "SVOA_SIM" as applicable.
LabDataPackageVersion	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabNarrative					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LabQualifiersDefinition	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	Report the Case Number.
ProjectName					Not required.
SiteID					Not required.
SiteName					Not required.
SamplePlusMethod	X	X	X	X	
Bottles					Not required.
BottleType					Not required.
ClientID	X	X			Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory	X	X	X		Report "PAH" for analyte subset when applicable.
ClientMethodCode	X	X	X		Report "PAH", "TCLP", or "SPLP" when applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	Report "GCMS_Internal_Standard".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
ClientName					Not required.
ClientSampleID	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X			Report the date and time the sample was collected.
CollectedEndDate					Not required.
Comment					Not required.
Composite					Not required.
CoolerID					Not required.
CustodyID	X	X			Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch					Not required.
Filtered					Not required.
LabContract	X	X	X		Report the Contract Number.
LabContractModificationDescription					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LabContractModificationID					Not required.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID					Not required.
LabMethodName					Not required.
LabName	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X			Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID					Not required.
LocationName					Not required.
MatrixID	X	X	X	X	Report "Water" or "Soil" as applicable.
MatrixMedium	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch					Not required.
MethodCategory					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X	X		Report "Trace", "Low", or "Medium".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	Report "GC/MS".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID	X	X			Report the EPA Sample Number of the original sample this sample was derived from. Report the EPA Sample Number used for the low level sample analysis for the volatiles and semivolatiles medium level samples, if applicable. Leave blank if only the medium level analysis is performed for the sample.
OriginalLabSampleID					Not required.
PhaseAnalyzed					Not required.
Preservative	X	X			Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X		Report the Case Number.
ProjectName					Not required.
QCCategory		X	X		Report "Blank" for MB, LEB, SB, or IB; "Spike" for MS; or "Spike_Duplicate" for MSD.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
QCLinkage		X	X		Report "LabReportingBatch" for MS/MSD, "PreparationBatch" for SVOA MB, "AnalysisBatch" for VOA IB, "StorageBatch" for SB, or "HandlingBatch" for LEB.
QCType	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Storage_Blank" for SB; "Method_Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; or "Non_Client_Sample".
Quarantine	X				Report "Yes" or "No" based on sampling information.
SamplingBatch					Not required.
ShippingBatch					Not required.
SiteID					Not required.
SiteName					Not required.
StorageBatch	X	X	X		Links all samples stored together with the Storage Blank. Report the Lab File ID of the Storage Blank. Not required for MB or IB.
InstrumentQC					Not required.
Characteristic	X	X	X		
CharacteristicType	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".
CharacteristicValue	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X		Report "C" for "Temperature".
Comment					Not required.
ContactInformation	X	X	X	X	
LabAddress1	X	X	X	X	Report the street address of the laboratory.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LabAddress2	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType					Not required.
LabState	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType					Not required.
LabZipCode	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
AnalysisBatch	X	X	X	X	Links this analysis to the instrument QC sample(s) that begins this sequence. Report the Lab File ID of the standard (Tune or CCV) that starts the sequence.
AnalysisBatchEnd	X	X	X	X	Links this analysis to the instrument QC sample(s) that ends this sequence. Report the Lab File ID of the CCV that ends this sequence.
AnalysisDuration					Not required.
AnalysisDurationUnits					Not required.
AnalysisGroupID					Not required.
AnalysisType	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.
Analyst	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X		For VOA medium soils/sediments, report the Soil Aliquot Volume in microliters to at least two significant figures.
AnalyzedAmountUnits	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
BackgroundRawData					Not required.
BackgroundType					Not required.
BottleID					Not required.
ClientAnalysisID	X	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode	X	X	X	X	Report "Full_Scan" or "SIM" as applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X		Report the GC column used.
ColumnInternalDiameter	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X		Report "mm".
ColumnLength	X	X	X		Report the Column Length in meters.
ColumnLengthUnits	X	X	X		Report "m".
Comment					Not required.
ConfirmationAnalysisID					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectorID					Not required.
DetectorType					Not required.
DilutionFactor	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency					Not required.
HeatedPurge	X	X	X		For VOA, report "Yes" if heated purge was used; otherwise report "No".
Inclusion					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
InjectionVolume	X	X	X		For VOA, report the purge volume in milliliters. For SVOA, report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X		Report "mL" or "uL" as applicable.
InstrumentID	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection					Not required.
LabAnalysisID	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	Report the Lab File ID.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
OriginalLabAnalysisID	X	X	X		If a dilution or reinjection is prepared from a previously analyzed sample, report the Lab File ID of the original sample from which the dilution or reinjection is prepared.
PreparationBatch					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
ReferenceDate					Not required.
ResultBasis	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
RunBatch	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL sequence.
SampleAmount					Not required.
SampleAmountUnits					Not required.
Temperature					Not required.
TemperatureUnits					Not required.
Wavelength					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
WavelengthUnits					Not required.
Yield					Not required.
AnalysisGroup					Not required.
Handling	X	X	X		
Analyst					Not required.
BottleID					Not required.
ClientMethodCode					Not required.
ClientMethodID	X	X	X		Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X		Report the month and year the SOW was issued.
Comment					Not required.
HandledDate	X	X	X		Enter the date and time TCLP or SPLP extraction began or decanting was performed.
HandlingBatch	X	X	X		Links all samples that were TCLP or SPLP extracted together or decanted together. Report a unique identifier for each batch.
HandlingType	X	X	X		Report "TCLP" or "SPLP" for extractions. Report "Decanted" if water was decanted from soil/sediment samples; otherwise report "Not_decanted".
InitialAmount					Not required.
InitialAmountUnits					Not required.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X		Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X		Report the month and year the SOW was issued.
ProcedureID					Not required.
ProcedureName					Not required.
SampleAmount					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
SampleAmountUnits					Not required.
ReportedResult	X	X	X		
AnalysisGroupID					Not required.
AnalyteGroupID					Not required.
AnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
AnalyteNameContext	X	X	X		Report "CAS" as applicable.
AnalyteType	X	X	X		Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD analysis, and "TIC" for all TICs.
BiasErrorRatio					Not required.
CASRegistryNumber	X	X	X		Report the CAS Number as it appears in the SOW, and for TICs if known.
ClientAnalyteID	X	X	X		Report the CAS number. For TICs with no CAS number, report TIC name or as "Unknown-01", then increment with each TIC.
ClientAnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
ClientDetectionLimit					Not required.
ClientDetectionLimitUnits					Not required.
ClientQuantitationLimit	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment					Not required.
DetectionLimit	X	X	X		For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio					Not required.
ExpectedResult		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits					Not required.
ExpectedResultUnits		X			Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
LabAnalysisID	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID					Not required.
LabQualifiers	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X			Report "Preliminary" or "Final" as applicable.
PeakID					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRecovery		X			Report the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh		X			Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow		X			Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X			Report "Method".
PercentRecoveryType					Not required.
QuantitationLimit	X	X	X		For target analytes, report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Result	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime	X	X	X		Report retention time in decimal minutes for all TICs.
RetentionTimeUnits	X	X	X		Report "Minutes".
RPD		X			Report the RPD to the nearest whole percent.
RPDLimitHigh		X			Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType		X			Report "Method".
RPDType					Not required.
PreparationPlusCleanup	X	X	X		
AliquotAmount	X	X	X		Report the sample amount in grams for soil/sediment or milliliters for aqueous/water (VOA and SVOA) to at least three significant figures.
AliquotAmountUnits	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X		Report the Analyst's initials.
BottleID					Not required.
CleanedUpDate	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X		Report "GPC" as applicable.
ClientMethodCode					Not required.
ClientMethodID	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X		Report the month and year the SOW was issued.
Comment					Not required.
Efficiency					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
FinalAmount	X	X	X		Report the Final Amount of material produced upon completion of this prep or cleanup in microliters (SVOA only).
FinalAmountUnits	X	X	X		Report "uL".
InitialAmount	X	X	X		Report the initial amount of extracted sample used for this prep or cleanup method in microliters (SVOA and Medium VOA soil/sediment).
InitialAmountUnits	X	X	X		Report "uL".
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
LotNumber					Not required.
MethodCode					Not required.
MethodID	X	X	X		Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X		Links all samples that were prepared together. Applicable to Trace VOA and VOA Low/Medium samples that were analyzed in the same analytical sequence. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Liq_Liq" or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution. Report "Purge_and_Trap" for Trace VOA and VOA Low/Medium.
PreparedDate	X	X	X		Report the date and time the sample was prepared or purged as applicable.
ProcedureID					Not required.
ProcedureName					Not required.
SampleAmount					Not required.
SampleAmountUnits					Not required.
Solvent					Not required.
Analyte	X	X	X		
AmountAdded	X	X	X		Volume of internal standard, DMC, or MS/MSD spiking solution added in microliters.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
AmountAddedUnits		X			Report "uL".
AmountAddedLocation		X			For MS/MSD, report "Aliquot".
AnalyteGroupID					Not required.
AnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment with each TIC.
AnalyteNameContext	X	X	X		Report "CAS" as applicable.
AnalyteType	X	X	X		Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD analysis, "Internal_Standard" for internal standards, "Surrogate" for DMCs, or "TIC" for all TICs.
BiasErrorRatio					Not required.
CalibrationBasis					Not required.
CalibrationFactor					Not required.
CalibrationFactorUnits					Not required.
CalibrationType					Not required.
CASRegistryNumber	X	X	X		Report the CAS Number as it appears in the SOW, and for TICs if known.
ClientAnalyteID	X	X	X		Report the CAS Number. For TICs with no CAS number, report TIC name or as "Unknown-01", then increment with each TIC.
ClientAnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment with each TIC.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectionLimit	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio					Not required.
Efficiency					Not required.
ExpectedResult	X	X	X		For DMCs and internal standards, report the final amount added in nanograms.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits					Not required.
ExpectedResultUnits	X	X	X		Report "ng".
Inclusion					Not required.
IntermediateResult	X	X	X		Report the on-column amount unadjusted for sample weight/volume, percent solids, or dilution factor, in nanograms, from the raw data. Leave blank if undetected.
IntermediateResultLimitHigh					Not required.
IntermediateResultLimitLow					Not required.
IntermediateResultLimitType					Not required.
IntermediateResultUnits	X	X	X		Report "ng".
LabAnalyteID					Not required.
LabQualifiers	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard (DMCs, internal standards, and spiking analytes only).
Mass					Not required.
MassLimitHigh					Not required.
MassLimitLow					Not required.
MassLimitType					Not required.
MassUnits					Not required.
MeanCalibrationFactor					Not required.
MeanCalibrationFactorUnits					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak. For unknown TICs, report the unique identifiers as applicable. For alkanes, report "Total alkanes" as the identifier.
PercentBreakdown					Not required.
PercentBreakdownLimitHigh					Not required.
PercentBreakdownLimitType					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentMatch	X		X		Report the percent match for TICs only.
PercentRecovery	X	X	X		Report the final calculated Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitHigh	X	X	X		Report the upper limit for the Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitLow	X	X	X		Report the lower limit for the Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitType	X	X	X		Report "Method".
PercentRecoveryType					Not required.
PercentRSD					Not required.
PercentRSDLimitHigh					Not required.
PercentRSDLimitLow					Not required.
PercentRSDLimitType					Not required.
QuantitationBasis					Not required.
QuantitationLimit	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Response					Not required.
ResponseLimitHigh					Not required.
ResponseLimitLow					Not required.
ResponseLimitType					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ResponseUnits					Not required.
Result	X	X	X		Report the final calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RPD					Not required.
RPDLimitHigh					Not required.
RPDLimitType					Not required.
RPDType					Not required.
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
StandardConcentration	X	X	X		Report the concentration of the internal standard, DMC, or spiking solution added to the sample in ug/L.
StandardConcentrationUnits					Report "ug/L".
StandardDeviation					Not required.
StandardDeviationUnits					Not required.
StandardFinalAmount					Not required.
StandardFinalAmountUnits					Not required.
StandardID					Not required.
StandardSource	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
WavelengthUnits					Not required.
WeightingFactor					Not required.
AnalyteComparison					Not required.
AnalyteGroup					Not required.
Peak	X	X	X		
CalibrationFactor					Not required.
CalibrationFactorUnits					Not required.
CalibrationType					Not required.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
DetectionLimit					Not required.
DetectionLimitType					Not required.
DetectionLimitUnits					Not required.
DifferenceErrorRatio					Not required.
Efficiency					Not required.
Inclusion					Not required.
IntermediateResult	X	X	X		Report the on-column amount in nanograms from the raw data. Leave blank if compound is not detected.
IntermediateResultLimitHigh					Not required.
IntermediateResultLimitLow					Not required.
IntermediateResultLimitType					Not required.
IntermediateResultUnits	X	X	X		Report "ng".
LabQualifiers					Not required.
ManualIntegration	X	X	X		Report "Yes" if this peak was manually integrated; otherwise report "No".
Mass					Not required.
MassLimitHigh					Not required.
MassLimitLow					Not required.
MassLimitType					Not required.
MassUnits					Not required.
MeanCalibrationFactor					Not required.
MeanCalibrationFactorUnits					Not required.
MeanRetentionTime					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
MeanRetentionTimeLimitHigh					Not required.
MeanRetentionTimeLimitLow					Not required.
MeanRetentionTimeLimitType					Not required.
MeanRetentionTimeUnits					Not required.
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X		Report the primary quantitation ion used or "Total" if all ions were used.
PeakRatio					Not required.
PeakRatioLimitHigh					Not required.
PeakRatioLimitLow					Not required.
PeakRatioLimitType					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRatio					Not required.
PercentRatioLimitHigh					Not required.
PercentRatioLimitLow					Not required.
PercentRatioLimitType					Not required.
PercentRecovery					Not required.
PercentRecoveryLimitHigh					Not required.
PercentRecoveryLimitLow					Not required.
PercentRecoveryLimitType					Not required.
PercentRecoveryType					Not required.
PercentRSD					Not required.
PercentRSDLimitHigh					Not required.
PercentRSDLimitLow					Not required.
PercentRSDLimitType					Not required.
QuantitationLimit					Not required.
QuantitationLimitType					Not required.
QuantitationLimitUnits					Not required.
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Resolution					Not required.
ResolutionLimitHigh					Not required.
ResolutionLimitLow					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ResolutionLimitType					Not required.
ResolutionType					Not required.
ResolutionUnits					Not required.
Response	X	X	X		Report the actual peak response from the raw data.
ResponseLimitHigh	X	X	X		Report the upper limit for the response for the internal standards only.
ResponseLimitLow	X	X	X		Report the lower limit for the response for the internal standards only.
ResponseLimitType	X	X	X		Report "Method".
ResponseType					Not required.
ResponseUnits	X	X	X		Report "Peak_Area".
Result					Not required.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType					Not required.
ResultUncertainty					Not required.
ResultUnits					Not required.
RetentionTime	X	X	X		Report the actual retention time in decimal minutes from the raw data for this peak.
RetentionTimeLimitHigh	X	X	X		Report the upper limit for this retention time in decimal minutes for the internal standards.
RetentionTimeLimitLow	X	X	X		Report the lower limit for this retention time in decimal minutes for the internal standards.
RetentionTimeLimitType	X	X	X		Report "Method".
RetentionTimeUnits	X	X	X		Report "Minutes".
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
StandardDeviation					Not required.
StandardDeviationUnits					Not required.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
PeakComparison	X	X	X		
AnalyteName	X	X	X		Report the name of the associated internal standard as it appears in the SOW.
AnalyteNameContext	X	X	X		Report "CAS".
CASRegistryNumber	X	X	X		Report the CAS number of the associated internal standard.
ClientAnalyteID	X	X	X		Report the CAS number of the associated internal standard.
ClientAnalyteName					Not required.
Comment					Not required.
LabAnalyteID					Not required.
PeakID	X	X	X		Report the primary quantitation ion used for the internal standard.
PeakRatio					Not required.
PeakRatioLimitHigh					Not required.
PeakRatioLimitLow					Not required.
PeakRatioLimitType					Not required.
PercentRatio					Not required.
PercentRatioLimitHigh					Not required.
PercentRatioLimitLow					Not required.
PercentRatioLimitType					Not required.
PeakReplicate					Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
Header	X	X	X	
ClientID	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName				Not required.
Comment				Not required.
DateFormat	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	Report the software version number.
LabContract	X	X	X	Report the Contract Number.
LabContractModificationDescription				Not required.
LabContractModificationID				Not required.
LabDataPackageID	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	Report "VOA_Trace", "VOA_Low_Med", "SVOA", or "SVOA_SIM" as applicable.
LabDataPackageVersion	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	Report the Lab Name.
LabNarrative				Not required.
LabQualifiersDefinition	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	Report the Case Number.
ProjectName				Not required.
SiteID				Not required.
SiteName				Not required.
SamplePlusMethod				Not required.
InstrumentQC	X	X	X	
ClientInstrumentQCType				Not required.
ClientMethodCode	X	X	X	Report "PAH", "TCLP", or "SPLP" when applicable.
ClientMethodID	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription				Not required.
ClientMethodModificationID	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName				Not required.
ClientMethodSource	X	X	X	Report "EPA_CLP".

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
ClientMethodVersion	X	X	X	Report the month and year the SOW was issued.
Comment				Not required.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	Report the EPA Sample Number. For ICAL, report the EPA Sample Number of the first standard.
LabMethodID				Not required.
LabMethodName				Not required.
LabName	X	X	X	Report the Lab Name.
MethodCode				Not required.
MethodID	X	X	X	Report "SOM02.4".
MethodModificationDescription				Not required.
MethodModificationID				Not required.
MethodName				Not required.
MethodSource	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	Report "RunBatch" for ICAL. Report "AnalysisBatch" for Tune and CCV.
QCType	X	X	X	Report "Instrument_Performance_Check_Tune" for Tune; "Initial_Calibration" for calibration; "Initial_Calibration_Verification" for ICV; or "Continuing_Calibration_Verification" for CCV.
ContactInformation	X	X	X	
LabAddress1	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	Report the title of the point of contact.
LabPointOfContactType				Not required.
LabState	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	Report the 10-digit phone number for the laboratory.
LabType				Not required.
LabZipCode	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	
AliquotAmount				Not required.
AliquotAmountUnits				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
AnalysisBatch			X	Links this analysis to the beginning of a 12-hour period. Report the Lab File ID of the standard (tune or CCV) that starts this sequence. For the standard that starts the 12-hour period, enter the Lab File ID of the standard itself.
AnalysisBatchEnd			X	Links this analysis to the end of a 12-hour period. Report the Lab File ID of the CCV that ends this sequence. For the closing CCV that closes the 12-hour period, report the Lab File ID of the standard itself.
AnalysisDuration				Not required.
AnalysisDurationUnits				Not required.
AnalysisGroupID		X		Links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard (Tune or calibration standard) that starts this ICAL sequence.
AnalysisType	X	X	X	For Tune, report "Initial". For ICAL/CCV, report the calibration level used.
Analyst	X	X	X	Report the Analyst's initials.
AnalyzedAmount				Not required.
AnalyzedAmountUnits				Not required.
AnalyzedDate	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection				Not required.
BackgroundRawData				Not required.
BackgroundType				Not required.
BottleID				Not required.
ClientAnalysisID	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode				Not required.
ClientMethodID	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription				Not required.
ClientMethodModificationID				Not required.
ClientMethodName				Not required.
ClientMethodSource	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	Report the GC Column used.
ColumnInternalDiameter	X	X	X	Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	Report "mm".
ColumnLength	X	X	X	Report the GC Column Length in meters.
ColumnLengthUnits	X	X	X	Report "m".
Comment				Not required.
ConfirmationAnalysisID				Not required.
Counts				Not required.
CountsUncertainty				Not required.
CountsUncertaintyConfidenceLevel				Not required.
CountsUncertaintyDetermination				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
CountsUncertaintyIntervalType				Not required.
CountsUncertaintyLimitHigh				Not required.
CountsUncertaintyLimitLow				Not required.
CountsUncertaintyType				Not required.
CountsUnits				Not required.
DetectorID				Not required.
DetectorType				Not required.
DilutionFactor	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency				Not required.
HeatedPurge	X	X	X	For VOA, report "Yes" if a heated purge was used; otherwise report "No".
Inclusion		X		Report "Yes" if the ICAL standard is to be included in the calibration curve; otherwise report "No".
InjectionVolume	X	X	X	For VOA, report the purge volume in milliliters. For SVOA, report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	Report "mL" or "uL" as applicable.
InstrumentID	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection				Not required.
LabAnalysisID	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	Report the Lab File ID.
LabID				Not required.
LabMethodID				Not required.
LabMethodName				Not required.
LabName				Not required.
MethodCode				Not required.
MethodID	X	X	X	Report "SOM02.4".
MethodModificationDescription				Not required.
MethodModificationID				Not required.
MethodName				Not required.
MethodSource	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	Report month and year the SOW was issued.
OriginalLabAnalysisID				Not required.
PreparationBatch				Not required.
ProcedureID				Not required.
ProcedureName				Not required.
ReferenceDate				Not required.
ResultBasis				Not required.
RunBatch	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard (Tune or calibration standard) that started the ICAL sequence.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
SampleAmount				Not required.
SampleAmountUnits				Not required.
Temperature				Not required.
TemperatureUnits				Not required.
Wavelength				Not required.
WavelengthUnits				Not required.
Yield				Not required.
AnalysisGroup		X		
AnalysisGroupID		X		This links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard (Tune or calibration) that starts this calibration sequence.
AnalysisType		X		Report "Initial_Calibration".
Comment				Not required.
Handling				Not required.
ReportedResult				Not required.
PreparationPlusCleanup				Not required.
Analyte	X	X	X	
AmountAdded	X	X	X	Report the volume of standard used in microliters.
AmountAddedUnits	X	X	X	Report "uL".
AmountAddedLocation	X	X	X	Report "Standard".
AnalyteGroupID				Not required.
AnalyteName	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	Report "CAS".
AnalyteType	X	X	X	Report "Target" for all target analytes, "Internal_Standard" for internal standards, "Surrogate" for DMCs, or "Instrument_Performance" for tunes.
BiasErrorRatio				Not required.
CalibrationBasis		X		Report "Peak" under the AnalysisGroup node.
CalibrationFactor				Not required.
CalibrationFactorUnits				Not required.
CalibrationType				Not required.
CASRegistryNumber	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	Report CAS Number.
ClientAnalyteName	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0				Not required.
Coeffa1				Not required.
Coeffa2				Not required.
Coeffa3				Not required.
CoeffOfDetermination				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
CoeffOfDeterminationLimitLow				Not required.
CoeffOfDeterminationLimitType				Not required.
Comment				Not required.
CorrelationCoeff				Not required.
CorrelationCoeffLimitLow				Not required.
CorrelationCoeffLimitType				Not required.
Counts				Not required.
CountsUncertainty				Not required.
CountsUncertaintyConfidenceLevel				Not required.
CountsUncertaintyDetermination				Not required.
CountsUncertaintyIntervalType				Not required.
CountsUncertaintyLimitHigh				Not required.
CountsUncertaintyLimitLow				Not required.
CountsUncertaintyType				Not required.
CountsUnits				Not required.
DetectionLimit				Not required.
DetectionLimitType				Not required.
DetectionLimitUnits				Not required.
DifferenceErrorRatio				Not required.
Efficiency				Not required.
ExpectedResult		X	X	Report the final amount for all applicable target analytes, DMCs, and internal standards.
ExpectedResultUncertainty				Not required.
ExpectedResultUncertaintyConfidenceLevel				Not required.
ExpectedResultUncertaintyDetermination				Not required.
ExpectedResultUncertaintyIntervalType				Not required.
ExpectedResultUncertaintyLimitHigh				Not required.
ExpectedResultUncertaintyLimitLow				Not required.
ExpectedResultUncertaintyType				Not required.
ExpectedResultUncertaintyUnits				Not required.
ExpectedResultUnits		X	X	Report "ng".
Inclusion		X		Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult		X	X	Report the on-column amount unadjusted for sample weight/volume, percent solids, or dilution factor, in nanograms, from the raw data.
IntermediateResultLimitHigh				Not required.
IntermediateResultLimitLow				Not required.
IntermediateResultLimitType				Not required.
IntermediateResultUnits		X	X	Report "ng".
LabAnalyteID				Not required.
LabQualifiers	X	X	X	Report qualifiers as specified in the SOW.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
LotNumber	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass				Not required.
MassLimitHigh				Not required.
MassLimitLow				Not required.
MassLimitType				Not required.
MassUnits				Not required.
MeanCalibrationFactor				Not required.
MeanCalibrationFactorUnits				Not required.
MeanRRF				Not required.
MeanRRFLimitLow				Not required.
MeanRRFLimitType				Not required.
PeakID		X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown				Not required.
PercentBreakdownLimitHigh				Not required.
PercentBreakdownLimitType				Not required.
PercentDifference				Not required.
PercentDifferenceLimitHigh				Not required.
PercentDifferenceLimitLow				Not required.
PercentDifferenceLimitType				Not required.
PercentMatch				Not required.
PercentRecovery				Not required.
PercentRecoveryLimitHigh				Not required.
PercentRecoveryLimitLow				Not required.
PercentRecoveryLimitType				Not required.
PercentRecoveryType				Not required.
PercentRSD				Not required.
PercentRSDLimitHigh				Not required.
PercentRSDLimitLow				Not required.
PercentRSDLimitType				Not required.
QuantitationBasis		X		Report "Internal_Standard" under the AnalysisGroup node.
QuantitationLimit				Not required.
QuantitationLimitType				Not required.
QuantitationLimitUnits				Not required.
ReportingLimit				Not required.
ReportingLimitType				Not required.
ReportingLimitUnits				Not required.
Response				Not required.
ResponseLimitHigh				Not required.
ResponseLimitLow				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
ResponseLimitType				Not required.
ResponseUnits				Not required.
Result				Not required.
ResultLimitHigh				Not required.
ResultLimitLow				Not required.
ResultLimitType				Not required.
ResultType				Not required.
ResultUncertainty				Not required.
ResultUncertaintyConfidenceLevel				Not required.
ResultUncertaintyDetermination				Not required.
ResultUncertaintyIntervalType				Not required.
ResultUncertaintyLimitHigh				Not required.
ResultUncertaintyLimitLow				Not required.
ResultUncertaintyType				Not required.
ResultUncertaintyUnits				Not required.
ResultUnits				Not required.
RPD				Not required.
RPDLimitHigh				Not required.
RPDLimitType				Not required.
RPDType				Not required.
RRF				Not required.
RRFLimitLow				Not required.
RRFLimitType				Not required.
StandardConcentration	X	X	X	Report the concentration of standard used in microgram per liter.
StandardConcentrationUnits	X	X	X	Report "ug/L".
StandardDeviation				Not required.
StandardDeviationUnits				Not required.
StandardFinalAmount				Not required.
StandardFinalAmountUnits				Not required.
StandardID	X	X	X	Report the laboratory-assigned identifier for this standard.
StandardSource	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor				Not required.
TailingFactorLimitHigh				Not required.
TailingFactorLimitType				Not required.
Wavelength				Not required.
WavelengthUnits				Not required.
WeightingFactor				Not required.
AnalyteComparison				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
AnalyteGroup				Not required.
Peak	X	X	X	
CalibrationFactor				Not required.
CalibrationFactorUnits				Not required.
CalibrationType		X		Report "Average_Response_Factor" under the AnalysisGroup node.
Coeffa0				Not required.
Coeffa1				Not required.
Coeffa2				Not required.
Coeffa3				Not required.
CoeffOfDetermination				Not required.
CoeffOfDeterminationLimitLow				Not required.
CoeffOfDeterminationLimitType				Not required.
Comment				Not required.
CorrelationCoeff				Not required.
CorrelationCoeffLimitLow				Not required.
CorrelationCoeffLimitType				Not required.
DetectionLimit				Not required.
DetectionLimitType				Not required.
DetectionLimitUnits				Not required.
DifferenceErrorRatio				Not required.
Efficiency				Not required.
Inclusion		X		Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult		X	X	Report the on-column in nanograms from the raw data.
IntermediateResultLimitHigh				Not required.
IntermediateResultLimitLow				Not required.
IntermediateResultLimitType				Not required.
IntermediateResultUnits		X	X	Report "ng".
LabQualifiers				Not required.
ManualIntegration	X	X	X	Report "Yes" if this peak was manually integrated; otherwise report "No".
Mass				Not required.
MassLimitHigh				Not required.
MassLimitLow				Not required.
MassLimitType				Not required.
MassUnits				Not required.
MeanCalibrationFactor				Not required.
MeanCalibrationFactorUnits				Not required.
MeanRetentionTime				Not required.
MeanRetentionTimeLimitHigh				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
MeanRetentionTimeLimitLow				Not required.
MeanRetentionTimeLimitType				Not required.
MeanRetentionTimeUnits				Not required.
MeanRRF		X		Report the calculated mean RRF to the nearest thousandth under the AnalysisGroup node only.
MeanRRFLimitLow				Not required.
MeanRRFLimitType				Not required.
PeakID	X	X	X	Report a unique identifier. This identifier must be consistent throughout an analytical sequence.
PeakRatio				Not required.
PeakRatioLimitHigh				Not required.
PeakRatioLimitLow				Not required.
PeakRatioLimitType				Not required.
PercentDifference			X	Report the calculated Percent Difference for this peak to the nearest tenth of a percent.
PercentDifferenceLimitHigh			X	Report the upper limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitLow			X	Report the lower limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitType			X	Report "Method".
PercentRatio				Not required.
PercentRatioLimitHigh				Not required.
PercentRatioLimitLow				Not required.
PercentRatioLimitType				Not required.
PercentRecovery				Not required.
PercentRecoveryLimitHigh				Not required.
PercentRecoveryLimitLow				Not required.
PercentRecoveryLimitType				Not required.
PercentRecoveryType				Not required.
PercentRSD		X		Report the calculated %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitHigh		X		Report the upper limit for the %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitLow				Not required.
PercentRSDLimitType		X		Report "Method".
QuantitationLimit				Not required.
QuantitationLimitType				Not required.
QuantitationLimitUnits				Not required.
ReportingLimit				Not required.
ReportingLimitType				Not required.
ReportingLimitUnits				Not required.
Resolution				Not required.
ResolutionLimitHigh				Not required.

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
ResolutionLimitLow				Not required.
ResolutionLimitType				Not required.
ResolutionType				Not required.
ResolutionUnits				Not required.
Response	X	X	X	Report the actual Peak Area from the raw data. For Tunes, report the abundance for the ion.
ResponseLimitHigh		X	X	Report the upper limit for this response for the internal standards only.
ResponseLimitLow		X	X	Report the lower limit for this response for the internal standards only.
ResponseLimitType		X	X	Report "Method".
ResponseType				Not required.
ResponseUnits	X	X	X	Report "Peak_Area" or "Abundance" as appropriate.
Result				Not required.
ResultLimitHigh				Not required.
ResultLimitLow				Not required.
ResultLimitType				Not required.
ResultType				Not required.
ResultUncertainty				Not required.
ResultUnits				Not required.
RetentionTime	X	X	X	Report the actual retention time in decimal minutes from the raw data for this peak.
RetentionTimeLimitHigh		X	X	Report the upper limit for this retention time in decimal minutes for the internal standards.
RetentionTimeLimitLow		X	X	Report the lower limit for this retention time in decimal minutes for the internal standards.
RetentionTimeLimitType		X	X	Report "Method".
RetentionTimeUnits	X	X	X	Report "Minutes".
RRF		X	X	Report the calculated RRF to the nearest thousandth.
RRFLimitLow		X	X	Report the lower limit for the RRF to the nearest thousandth.
RRFLimitType		X	X	Report "Method".
StandardDeviation				Not required.
StandardDeviationUnits				Not required.
TailingFactor				Not required.
TailingFactorLimitHigh				Not required.
TailingFactorLimitType				Not required.
Wavelength				Not required.
WavelengthUnits				Not required.
WeightingFactor				Not required.
PeakComparison	X	X	X	
AnalyteName	X	X	X	Report the tune compound or the associated internal standard as they appear in the SOW.
AnalyteNameContext	X	X	X	Report "CAS".

TABLE 1. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	Applicability: ICAL	Applicability: ICV/CCV	Instructions
CASRegistryNumber	X	X	X	Report the CAS number of the tune compound or associated internal standard.
ClientAnalyteID	X	X	X	Report the CAS number of the tune compound or associated internal standard.
ClientAnalyteName				Not required.
Comment				Not required.
PeakID	X	X	X	For tunes, report the mass being compared to the monitored mass. For internal standards, report the primary quantitation ion.
PeakRatio				Not required.
PeakRatioLimitHigh				Not required.
PeakRatioLimitLow				Not required.
PeakRatioLimitType				Not required.
PercentRatio	X			Report the Percent Ratio (%Relative Abundance or %Mass) to the nearest hundredth.
PercentRatioLimitHigh	X			Report the upper limit for the Percent Ratio to the nearest hundredth.
PercentRatioLimitLow	X			Report the lower limit for the Percent Ratio to the nearest hundredth.
PercentRatioLimitType	X			Report "Method".
PeakReplicate				Not required.

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TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Header	X	X	X	X	
ClientID	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName					Not required.
Comment					Not required.
DateFormat	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabDataPackageID	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	Report "VOA_Trace", "VOA_Low_Med", "SVOA", or "SVOA_SIM" as applicable.
LabDataPackageVersion	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabNarrative					Not required.
LabQualifiersDefinition	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	Report the Case Number.
ProjectName					Not required.
SiteID					Not required.
SiteName					Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	Sample	MS/MSD	MB/LEB/SB/IB	NCS	
SamplePlusMethod	X	X	X	X	
ClientID	X	X			Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory	X	X	X		Report "PAH" for analyte subset when applicable.
ClientMethodCode	X	X	X		Report "PAH", "TCLP", or "SPLP" when applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	Report "GCMS_Internal_Standard".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
ClientName					Not required.
ClientSampleID	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X			Report the date and time the sample was collected.
CollectedEndDate					Not required.
Comment					Not required.
Composite					Not required.
CoolerID					Not required.
CustodyID	X	X			Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch					Not required.
Filtered					Not required.
LabContract	X	X	X		Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID					Not required.
LabMethodName					Not required.
LabName	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X			Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LocationID					Not required.
LocationName					Not required.
MatrixID	X	X	X	X	Report "Water" or "Soil" as applicable.
MatrixMedium	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch					Not required.
MethodCategory					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X	X		Report "Trace", "Low", or "Medium".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	Report "GC/MS".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID	X	X			Report the EPA Sample Number of the original sample this sample was derived from. Report the EPA Sample Number used for the low level sample analysis for the volatiles and semivolatiles medium level samples, if applicable. Leave blank if only the medium level analysis is performed for the sample.
OriginalLabSampleID					Not required.
PhaseAnalyzed					Not required.
Preservative	X	X			Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X		Report the Case Number.
ProjectName					Not required.
QCCategory		X	X		Report "Blank" for MB, LEB, SB, or IB; "Spike" for MS; or "Spike_Duplicate" for MSD.
QCLinkage		X	X		Report "LabReportingBatch" for MS/MSD, "PreparationBatch" for SVOA MB, "AnalysisBatch" for VOA IB, or "StorageBatch" for SB.
QCType	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Storage_Blank" for SB; "Method_Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; or "Non_Client_Sample".

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Quarantine	X				Report "Yes" or "No" based on sampling information.
SamplingBatch					Not required.
ShippingBatch					Not required.
SiteID					Not required.
SiteName					Not required.
StorageBatch	X	X	X		Links all samples stored together with the Storage Blank. Report the Lab File ID of the Storage Blank. Not required for MB or IB.
InstrumentQC					Not required.
Characteristic	X	X	X		
CharacteristicType	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".
CharacteristicValue	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X		Report "C" for "Temperature".
Comment					Not required.
ContactInformation	X	X	X	X	
LabAddress1	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	Report the Email address of the point of contact.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LabPointOfContactTitle	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType					Not required.
LabState	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType					Not required.
LabZipCode	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
AnalysisBatch	X	X	X	X	Links this analysis to the instrument QC sample(s) that begins this sequence. Report the Lab File ID of the standard (Tune or CCV) that starts the sequence.
AnalysisBatchEnd	X	X	X	X	Links this analysis to the instrument QC sample(s) that ends this sequence. Report the Lab File ID of the CCV that ends this sequence.
AnalysisDuration					Not required.
AnalysisDurationUnits					Not required.
AnalysisGroupID					Not required.
AnalysisType	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.
Analyst	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X		For VOA medium soils/sediments, report the Soil Aliquot Volume in microliters to at least 2 significant figures.
AnalyzedAmountUnits	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X		Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode	X	X	X	X	Report "Full_Scan" or "SIM" as applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X		Report the GC Column used.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ColumnInternalDiameter	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X		Report "mm".
ColumnLength	X	X	X		Report the Column Length in meters.
ColumnLengthUnits	X	X	X		Report "m".
Comment					Not required.
ConfirmationAnalysisID					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectorID					Not required.
DetectorType					Not required.
DilutionFactor	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency					Not required.
HeatedPurge	X	X	X		For VOA, report "Yes" if heated purge was used; otherwise report "No".
Inclusion					Not required.
InjectionVolume	X	X	X		For VOA, report the purge volume in milliliters. For SVOA, report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X		Report "mL" or "uL" as applicable.
InstrumentID	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	Report the Lab File ID.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
ReferenceDate					Not required.
ResultBasis	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
RunBatch	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL sequence.
Temperature					Not required.
TemperatureUnits					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
Yield					Not required.
AnalysisGroup					Not required.
Handling					Not required.
ReportedResult	X	X	X		
AnalysisGroupID					Not required.
AnalyteGroupID					Not required.
AnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
AnalyteNameContext	X	X	X		Report "CAS" as applicable.
AnalyteType	X	X	X		Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD analysis, and "TIC" for all TICs.
BiasErrorRatio					Not required.
CASRegistryNumber	X	X	X		Report the CAS Numbers as they appear in the SOW, and for TICs if known.
ClientAnalyteID	X	X	X		Report the CAS Number. For TICs with no CAS number, report TIC name or as "Unknown-01", then increment for each TIC.
ClientAnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ClientDetectionLimit					Not required.
ClientDetectionLimitUnits					Not required.
ClientQuantitationLimit	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment					Not required.
DetectionLimit	X	X	X		For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio					Not required.
ExpectedResult		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits					Not required.
ExpectedResultUnits		X			Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water.
LabAnalysisID	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID					Not required.
LabQualifiers	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X			Report "Preliminary" or "Final" as applicable.
PeakID					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRecovery		X			Report the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh		X			Report the upper limit for the Percent Recovery to the nearest whole percent.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
PercentRecoveryLimitLow		X			Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X			Report "Method".
PercentRecoveryType					Not required.
QuantitationLimit	X	X	X		Report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Result	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime	X	X	X		Report the retention time in decimal minutes for all TICs.
RetentionTimeUnits	X	X	X		Report "Minutes".
RPD		X			Report the RPD to the nearest whole percent.
RPDLimitHigh		X			Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType		X			Report "Method".
RPDType					Not required.
PreparationPlusCleanup	X	X	X		
AliquotAmount	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water (VOA and SVOA) to at least three significant figures.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
AliquotAmountUnits	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X		Report the Analyst's initials.
CleanedUpDate	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X		Report "GPC" as applicable.
ClientMethodCode					Not required.
ClientMethodID	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X		Report the month and year the SOW was issued.
Comment					Not required.
FinalAmount	X	X	X		Report the Final Amount of material produced upon completion of this prep or cleanup in microliters (SVOA only).
FinalAmountUnits	X	X	X		Report "uL".
InitialAmount	X	X	X		Report the initial amount of extracted sample used for this prep or cleanup method in microliters (SVOA and Medium VOA soil/sediment).
InitialAmountUnits	X	X	X		Report "uL".
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
LotNumber					Not required.
MethodCode					Not required.
MethodID	X	X	X		Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X		Report the month and year the SOW was issued.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
PreparationBatch	X	X	X		Links all samples that were prepared together. Applicable to Trace VOA and VOA Low/Medium samples that were analyzed in the same analytical sequence. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Liq_Liq" or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution. Report "Purge_and_Trap" for Trace VOA and VOA Low/Medium.
PreparedDate	X	X	X		Report the date and time the sample was prepared or purged as applicable.
ProcedureID					Not required.
ProcedureName					Not required.
Solvent					Not required.
Analyte	X	X	X		
AnalyteGroupID					Not required.
AnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment with each TIC.
AnalyteNameContext	X	X	X		Report "CAS" as applicable.
AnalyteType	X	X	X		Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD, "Internal_Standard" for internal standards, "Surrogate" for DMCs, or "TIC" for all TICs.
BiasErrorRatio					Not required.
CalibrationBasis					Not required.
CalibrationFactor					Not required.
CalibrationFactorUnits					Not required.
CalibrationType					Not required.
CASRegistryNumber	X	X	X		Report the CAS Number as it appears in the SOW, and for TIC if known.
ClientAnalyteID	X	X	X		Report the CAS Number. For TICs with no CAS Number, report TIC name or as "Unknown-01", then increment for each TIC.
ClientAnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment with each TIC.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectionLimit	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio					Not required.
Efficiency					Not required.
ExpectedResult	X	X	X		For DMCs and internal standards, report the final amount added in nanograms.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits	X	X	X		Report "ng".
ExpectedResultUnits					Not required.
Inclusion					Not required.
LabAnalyteID					Not required.
LabQualifiers	X	X	X		Report the qualifiers as specified in the SOW.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
LotNumber	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard (DMCs, internal standards, and spiking analytes only).
Mass					Not required.
MassUnits					Not required.
MeanCalibrationFactor					Not required.
MeanCalibrationFactorUnits					Not required.
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak. For unknown TICs, report the unique identifiers as applicable. For alkanes, report "Total alkanes" as the identifier.
PercentBreakdown					Not required.
PercentBreakdownLimitHigh					Not required.
PercentBreakdownLimitType					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRecovery	X	X	X		Report the final calculated Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitHigh	X	X	X		Report the upper limit for the Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitLow	X	X	X		Report the lower limit of the Percent Recovery of the DMCs to the nearest whole percent.
PercentRecoveryLimitType	X	X	X		Report "Method".
PercentRecoveryType					Not required.
PercentRSD					Not required.
PercentRSDLimitHigh					Not required.
PercentRSDLimitLow					Not required.
PercentRSDLimitType					Not required.
QuantitationBasis					Not required.
QuantitationLimit	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ReportingLimitUnits					Not required.
Result	X	X	X		Report the final calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RPD					Not required.
RPDLimitHigh					Not required.
RPDLimitType					Not required.
RPDType					Not required.
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
StandardSource	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.
AnalyteGroup					Not required.
Peak					Not required.
PeakComparison					Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
Header	X	X	X	
ClientID	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName				Not required.
Comment				Not required.
DateFormat	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	Report the software version number.
LabContract	X	X	X	Report the Contract Number.
LabContractModificationDescription				Not required.
LabContractModificationID				Not required.
LabDataPackageID	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	Report "VOA_Trace", "VOA_Low_Med", "SVOA", or "SVOA_SIM" as applicable.
LabDataPackageVersion	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	Report the Lab Name.
LabNarrative				Not required.
LabQualifiersDefinition	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	Report the Case Number.
ProjectName				Not required.
SiteID				Not required.
SiteName				Not required.
SamplePlusMethod				Not required.
InstrumentQC	X	X	X	
ClientInstrumentQCType				Not required.
ClientMethodCode	X	X	X	Report "PAH", "TCLP", or "SPLP" when applicable.
ClientMethodID	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription				Not required.
ClientMethodModificationID	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName				Not required.
ClientMethodSource	X	X	X	Report "EPA_CLP".

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
ClientMethodVersion	X	X	X	Report the month and year the SOW was issued.
Comment				Not required.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	Report the EPA Sample Number. For ICAL, report the EPA Sample Number of the first standard.
LabMethodID				Not required.
LabMethodName				Not required.
LabName	X	X	X	Report the Lab Name.
MethodCode				Not required.
MethodID	X	X	X	Report "SOM02.4".
MethodModificationDescription				Not required.
MethodModificationID				Not required.
MethodName				Not required.
MethodSource	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	Report "RunBatch" for ICAL. Report "AnalysisBatch" for Tune and CCV.
QCType	X	X	X	Report "Instrument_Performance_Check_Tune" for Tune; "Initial_Calibration" for calibration; "Initial_Calibration_Verification" for ICV; or "Continuing_Calibration_Verification" for CCV.
ContactInformation	X	X	X	
LabAddress1	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	Report the title of the point of contact.
LabPointOfContactType				Not required.
LabState	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	Report the 10-digit phone number for the laboratory.
LabType				Not required.
LabZipCode	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	
AliquotAmount				Not required.
AliquotAmountUnits				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
AnalysisBatch			X	Links this analysis to the beginning of a 12-hour period. Report the Lab File ID of the standard (Tune or CCV) that starts this sequence. For the standard that starts the 12-hour period, enter the Lab File ID of the standard itself.
AnalysisBatchEnd			X	Links this analysis to the end of a 12-hour period. Report the Lab File ID of the CCV that ends this sequence. For the closing CCV that closes the 12-hour period, report the Lab File ID of the standard itself.
AnalysisDuration				Not required.
AnalysisDurationUnits				Not required.
AnalysisGroupID		X		Links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard (Tune or calibration standard) that starts this ICAL sequence.
AnalysisType	X	X	X	For Tune, report "Initial". For ICAL/CCV, report the calibration level used.
Analyst	X	X	X	Report the Analyst's initials.
AnalyzedAmount				Not required.
AnalyzedAmountUnits				Not required.
AnalyzedDate	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode				Not required.
ClientMethodID	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription				Not required.
ClientMethodModificationID				Not required.
ClientMethodName				Not required.
ClientMethodSource	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	Report the GC Column used.
ColumnInternalDiameter	X	X	X	Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	Report "mm".
ColumnLength	X	X	X	Report the GC Column Length in meters.
ColumnLengthUnits	X	X	X	Report "m".
Comment				Not required.
ConfirmationAnalysisID				Not required.
Counts				Not required.
CountsUncertainty				Not required.
CountsUncertaintyConfidenceLevel				Not required.
CountsUncertaintyDetermination				Not required.
CountsUncertaintyIntervalType				Not required.
CountsUncertaintyLimitHigh				Not required.
CountsUncertaintyLimitLow				Not required.
CountsUncertaintyType				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
CountsUnits				Not required.
DetectorID				Not required.
DetectorType				Not required.
DilutionFactor	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency				Not required.
HeatedPurge	X	X	X	For VOA, report "Yes" if heated purge was used; otherwise report "No".
Inclusion	X	X	X	Report "Yes" if the ICAL standard is to be included in the calibration curve; otherwise report "No".
InjectionVolume	X	X	X	For VOA, report the purge volume in milliliters. For SVOA, report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	Report "mL" or "uL" as applicable.
InstrumentID	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	Report the Lab File ID.
LabID				Not required.
LabMethodID				Not required.
LabMethodName				Not required.
LabName				Not required.
MethodCode				Not required.
MethodID	X	X	X	Report "SOM02.4".
MethodModificationDescription				Not required.
MethodModificationID				Not required.
MethodName				Not required.
MethodSource	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	Report the month and year the SOW was issued.
PreparationBatch				Not required.
ProcedureID				Not required.
ProcedureName				Not required.
ReferenceDate				Not required.
ResultBasis				Not required.
RunBatch	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard (Tune or calibration standard) that started the ICAL sequence.
Temperature				Not required.
TemperatureUnits				Not required.
Wavelength				Not required.
WavelengthUnits				Not required.
Yield				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
AnalysisGroup		X		
AnalysisGroupID		X		This links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard that starts this calibration sequence.
AnalysisType		X		Report "Initial_Calibration".
Comment				Not required.
Handling				Not required.
ReportedResult				Not required.
PreparationPlusCleanup				Not required.
Analyte	X	X	X	
AnalyteGroupID				Not required.
AnalyteName	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	Report "CAS".
AnalyteType	X	X	X	Report "Target" for all target analytes, "Internal_Standard" for internal standards, "Surrogate" for DMCs, or "Instrument_Performance" for tunes.
BiasErrorRatio				Not required.
CalibrationBasis		X		Report "Peak" under the AnalysisGroup node.
CalibrationFactor				Not required.
CalibrationFactorUnits				Not required.
CalibrationType				Not required.
CASRegistryNumber	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	Report CAS Number.
ClientAnalyteName	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0				Not required.
Coeffa1				Not required.
Coeffa2				Not required.
Coeffa3				Not required.
CoeffOfDetermination				Not required.
CoeffOfDeterminationLimitLow				Not required.
CoeffOfDeterminationLimitType				Not required.
Comment				Not required.
CorrelationCoeff				Not required.
CorrelationCoeffLimitLow				Not required.
CorrelationCoeffLimitType				Not required.
Counts				Not required.
CountsUncertainty				Not required.
CountsUncertaintyConfidenceLevel				Not required.
CountsUncertaintyDetermination				Not required.
CountsUncertaintyIntervalType				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
CountsUncertaintyLimitHigh				Not required.
CountsUncertaintyLimitLow				Not required.
CountsUncertaintyType				Not required.
CountsUnits				Not required.
DetectionLimit				Not required.
DetectionLimitType				Not required.
DetectionLimitUnits				Not required.
DifferenceErrorRatio				Not required.
Efficiency				Not required.
ExpectedResult		X	X	Report the final amount for all applicable target analytes, DMCs, and internal standards.
ExpectedResultUncertainty				Not required.
ExpectedResultUncertaintyConfidenceLevel				Not required.
ExpectedResultUncertaintyDetermination				Not required.
ExpectedResultUncertaintyIntervalType				Not required.
ExpectedResultUncertaintyLimitHigh				Not required.
ExpectedResultUncertaintyLimitLow				Not required.
ExpectedResultUncertaintyType				Not required.
ExpectedResultUncertaintyUnits				Not required.
ExpectedResultUnits		X	X	Report "ng".
Inclusion		X		Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabAnalyteID				Not required.
LabQualifiers	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass				Not required.
MassUnits				Not required.
MeanCalibrationFactor				Not required.
MeanCalibrationFactorUnits				Not required.
MeanRRF				Not required.
MeanRRFLimitLow				Not required.
MeanRRFLimitType				Not required.
PeakID		X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown				Not required.
PercentBreakdownLimitHigh				Not required.
PercentBreakdownLimitType				Not required.
PercentDifference				Not required.
PercentDifferenceLimitHigh				Not required.
PercentDifferenceLimitLow				Not required.
PercentDifferenceLimitType				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
PercentRecovery				Not required.
PercentRecoveryLimitHigh				Not required.
PercentRecoveryLimitLow				Not required.
PercentRecoveryLimitType				Not required.
PercentRecoveryType				Not required.
PercentRSD				Not required.
PercentRSDLimitHigh				Not required.
PercentRSDLimitLow				Not required.
PercentRSDLimitType				Not required.
QuantitationBasis		X		Report "Internal_Standard" under the AnalysisGroup node.
QuantitationLimit				Not required.
QuantitationLimitType				Not required.
QuantitationLimitUnits				Not required.
ReportingLimit				Not required.
ReportingLimitType				Not required.
ReportingLimitUnits				Not required.
Result				Not required.
ResultLimitHigh				Not required.
ResultLimitLow				Not required.
ResultLimitType				Not required.
ResultType				Not required.
ResultUncertainty				Not required.
ResultUncertaintyConfidenceLevel				Not required.
ResultUncertaintyDetermination				Not required.
ResultUncertaintyIntervalType				Not required.
ResultUncertaintyLimitHigh				Not required.
ResultUncertaintyLimitLow				Not required.
ResultUncertaintyType				Not required.
ResultUncertaintyUnits				Not required.
ResultUnits				Not required.
RPD				Not required.
RPDLimitHigh				Not required.
RPDLimitType				Not required.
RPDType				Not required.
RRF				Not required.
RRFLimitLow				Not required.
RRFLimitType				Not required.
StandardSource	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor				Not required.
TailingFactorLimitHigh				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
TailingFactorLimitType				Not required.
Wavelength				Not required.
WavelengthUnits				Not required.
WeightingFactor				Not required.
AnalyteGroup				Not required.
Peak	X	X	X	
CalibrationFactor				Not required.
CalibrationFactorUnits				Not required.
CalibrationType		X		Report "Average_Response_Factor" under the AnalysisGroup node.
Coeffa0				Not required.
Coeffa1				Not required.
Coeffa2				Not required.
Coeffa3				Not required.
CoeffOfDetermination				Not required.
CoeffOfDeterminationLimitLow				Not required.
CoeffOfDeterminationLimitType				Not required.
Comment				Not required.
CorrelationCoeff				Not required.
CorrelationCoeffLimitLow				Not required.
CorrelationCoeffLimitType				Not required.
DifferenceErrorRatio				Not required.
Efficiency				Not required.
Inclusion		X		Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabQualifiers				Not required.
Mass				Not required.
MassLimitHigh				Not required.
MassLimitLow				Not required.
MassLimitType				Not required.
MassUnits				Not required.
MeanCalibrationFactor				Not required.
MeanCalibrationFactorUnits				Not required.
MeanRetentionTime				Not required.
MeanRetentionTimeLimitHigh				Not required.
MeanRetentionTimeLimitLow				Not required.
MeanRetentionTimeLimitType				Not required.
MeanRetentionTimeUnits				Not required.
MeanRRF		X		Report the calculated mean RRF to the nearest thousandth under the AnalysisGroup only.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
MeanRRFLimitLow				Not required.
MeanRRFLimitType				Not required.
PeakID	X	X	X	Report a unique identifier. This identifier must be consistent throughout the analytical sequence.
PercentDifference			X	Report the calculated Percent Difference for this peak to the nearest tenth of a percent.
PercentDifferenceLimitHigh			X	Report the upper limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitLow			X	Report the lower limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitType			X	Report "Method".
PercentRecovery				Not required.
PercentRecoveryLimitHigh				Not required.
PercentRecoveryLimitLow				Not required.
PercentRecoveryLimitType				Not required.
PercentRecoveryType				Not required.
PercentRSD		X		Report the calculated Percent Relative Standard Deviation to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitHigh		X		Report the upper limit for the Percent Relative Standard Deviation to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitLow				Not required.
PercentRSDLimitType		X		Report "Method".
Resolution				Not required.
ResolutionLimitHigh				Not required.
ResolutionLimitLow				Not required.
ResolutionLimitType				Not required.
ResolutionType				Not required.
ResolutionUnits				Not required.
Result				Not required.
ResultLimitHigh				Not required.
ResultLimitLow				Not required.
ResultLimitType				Not required.
ResultType				Not required.
ResultUncertainty				Not required.
ResultUnits				Not required.
RRF				Not required.
RRFLimitLow				Not required.
RRFLimitType				Not required.
TailingFactor				Not required.
TailingFactorLimitHigh				Not required.
TailingFactorLimitType				Not required.
Wavelength				Not required.

TABLE 2. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Tune	ICAL	ICV/CCV	Instructions
WavelengthUnits				Not required.
WeightingFactor				Not required.
PeakComparison	X	X	X	
Comment				Not required.
PeakID	X	X	X	For tunes, report the mass being compared to the monitored mass. For internal standards, report the primary quantitation ion.
PercentRatio	X			Report the Percent Ratio (%Relative Abundance or %Mass) to the nearest hundredth.
PercentRatioLimitHigh	X			Report the upper limit for the Percent Ratio to the nearest hundredth.
PercentRatioLimitLow	X			Report the lower limit for the Percent Ratio to the nearest hundredth.
PercentRatioLimitType	X			Report "Method".

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TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Header	X	X	X	X	
ClientID	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName					Not required.
Comment					Not required.
DateFormat	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	Report "SEDD_5-2_GENERAL_2a" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	Report "2" (This is the version of the DTD used).
EDDVersion	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	Report the software version number.
Lab Contract	X	X	X	X	Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabDataPackageID	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	Report "VOA_Trace", "VOA_Low_Med", "SVOA", or "SVOA_SIM" as applicable.
LabDataPackageVersion	X	X	X	X	Report "1", then increment with each resubmission.
LabID					Report the Agency-assigned Lab Code.
Lab Name	X	X	X	X	Report the Lab Name.
LabNarrative					Not required.
LabQualifiersDefinition	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	Report the Case Number.
ProjectName					Not required.
SiteID					Not required.
SiteName					Not required.
SamplePlusMethod	X	X	X	X	
ClientID	X	X			Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ClientMethodCategory	X	X	X	X	Report "PAH" for analyte subset when applicable.
ClientMethodCode	X	X	X		Report "PAH", "TCLP", or "SPLP" when applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	Report "GCMS_Internal_Standard".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
ClientName					Not required.
ClientSampleID	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X			Report the date and time the sample was collected.
CollectedEndDate					Not required.
Comment					Not required.
Composite					Not required.
CoolerID					Not required.
CustodyID	X	X			Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch					Not required.
Filtered					Not required.
LabContract	X	X	X		Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID					Not required.
LabMethodName					Not required.
LabName	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X			Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID					Not required.
LocationName					Not required.
MatrixID	X	X	X	X	Report "Water" or "Soil" as applicable.
MatrixMedium	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch					Not required.
MethodCategory					Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X			Report "Trace", "Low", or "Medium".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	Report "GC/MS".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID	X	X			Report the EPA Sample Number of the original sample this sample was derived from. Report the EPA Sample Number used for the low level sample analysis for the volatiles and semivolatiles medium level samples, if applicable. Leave blank if only the medium level analysis is performed for the sample.
OriginalLabSampleID					Not required.
PhaseAnalyzed					Not required.
Preservative	X	X			Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X		Report the Case Number.
ProjectName					Not required.
QCCategory		X	X		Report "Blank" for MB, LEB, SB, or IB; "Spike" for MS; or "Spike_Duplicate" for MSD.
QCLinkage		X	X		Report "LabReportingBatch" for MS/MSD, "PreparationBatch" for SVOA MB, "AnalysisBatch" for VOA IB, or "StorageBatch" for SB.
QCType	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Storage_Blank" for SB; "Method_Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; or "Non_Client_Sample".
Quarantine	X				Report "Yes" or "No" based on sampling information.
SamplingBatch					Not required.
ShippingBatch					Not required.
SiteID					Not required.
SiteName					Not required.
StorageBatch	X	X	X		List all samples stored together with the Storage Blank. Report the Lab File ID of the Storage Blank. Not required for MB or IB.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
Characteristic	X	X	X		
CharacteristicType	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".
CharacteristicValue	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X		Report "C" for "Temperature".
Comment					Not required.
ContactInformation	X	X	X	X	
LabAddress1	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType					Not required.
LabState	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType					Not required.
LabZipCode	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
AnalysisDuration					Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
AnalysisDurationUnits					Not required.
AnalysisGroupID					Not required.
AnalysisType	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.
Analyst	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X		For VOA medium soils/sediments, report the Soil Aliquot Volume in microliters to at least two significant figures.
AnalyzedAmountUnits	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X		Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode	X	X	X		Report "Full_Scan" or "SIM" as applicable.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report month and year the SOW was issued.
Column	X	X	X		Report the GC Column used.
ColumnInternalDiameter	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X		Report the Column Length in meters.
ColumnLength	X	X	X		Report "m".
ColumnLengthUnits					Not required.
Comment					Not required.
ConfirmationAnalysisID					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectorID					Not required.
DetectorType					Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
DilutionFactor	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency					Not required.
HeatedPurge	X	X	X		For VOA, report "Yes" if heated purge was used; otherwise report "No".
Inclusion					Not required.
InjectionVolume	X	X	X		For VOA, report the purge volume in milliliters. For SVOA, report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X		Report "mL" or "uL" as applicable.
InstrumentID	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	Report the Lab File ID.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
ReferenceDate					Not required.
ResultBasis	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
Temperature					Not required.
TemperatureUnits					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
Yield					Not required.
AnalysisGroup					Not required.
Handling					Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB NCS	Instructions
ReportedResult	X	X	X	
AnalysisGroupID				Not required.
AnalyteGroupID				Not required.
AnalyteName	X	X	X	Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
AnalyteNameContext	X	X	X	Report "CAS" as applicable.
AnalyteType	X	X	X	Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD analysis, and "TIC" for all TICs.
BiasErrorRatio				Not required.
CASRegistryNumber	X	X	X	Report the CAS Numbers as it appears in the SOW, and for TICs if known.
ClientAnalyteID	X	X	X	Report the CAS Number. For TICs with no CAS number, report TIC name or as "Unknown-01", then increment for each TIC.
ClientAnalyteName	X	X	X	Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
ClientDetectionLimit				Not required.
ClientDetectionLimitUnits				Not required.
ClientQuantitationLimit	X	X	X	Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment				Not required.
DetectionLimit	X	X	X	For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X	Report "MDL_sa".
DetectionLimitUnits	X	X	X	Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio				Not required.
ExpectedResult		X		Report the theoretical final calculated concentration (the spike added) for the spiked analytes.
ExpectedResultUncertainty				Not required.
ExpectedResultUncertaintyConfidenceLevel				Not required.
ExpectedResultUncertaintyDetermination				Not required.
ExpectedResultUncertaintyIntervalType				Not required.
ExpectedResultUncertaintyLimitHigh				Not required.
ExpectedResultUncertaintyLimitLow				Not required.
ExpectedResultUncertaintyType				Not required.
ExpectedResultUncertaintyUnits				Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ExpectedResultUnits		X			Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
LabAnalysisID	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID					Not required.
LabQualifiers	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X			Report "Preliminary" or "Final" as applicable.
PeakID					Not required.
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRecovery		X			Report the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh		X			Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow		X			Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X			Report "Method".
PercentRecoveryType					Not required.
QuantitationLimit	X	X	X		For target analytes, report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Result	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime	X	X	X		Report the retention time in decimal minutes for all TICs.
RetentionTimeUnits	X	X	X		Report "Minutes".
RPD		X			Report the RPD to the nearest whole percent.
RPDLimitHigh		X			Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType		X			Report "Method".
RPDType					Not required.
PreparationPlusCleanup	X	X	X		
AliquotAmount	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water (VOA and SVOA) to at least three significant figures.
AliquotAmountUnits	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X		Report the Analyst's initials.
CleanedUpDate	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X		Report "GPC" as applicable.
ClientMethodCode					Not required.
ClientMethodID	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X		Report the month and year the SOW was issued.
Comment					Not required.
FinalAmount	X	X	X		Report the volume of material produced upon completion of this Prep or Cleanup in microliters (SVOA only).
FinalAmountUnits	X	X	X		Report "uL".
InitialAmount	X	X	X		Report the initial amount of extracted sample used for this prep or cleanup method in microliters (SVOA and Medium VOA soil/sediment).

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
InitialAmountUnits	X	X	X		Report "uL".
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
LotNumber					Not required.
MethodCode					Not required.
MethodID	X	X	X		Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X		Links all samples that were prepared together. Applicable to Trace VOA and VOA Low/Medium samples that were analyzed in the same analytical sequence. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Liq_Liq" or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution. Report "Purge_and_Trap" for Trace VOA and VOA Low/Medium.
PreparedDate	X	X	X		Report the date and time the sample was prepared or purged as applicable.
ProcedureID					Not required.
ProcedureName					Not required.
Solvent					Not required.
Analyte	X	X	X		
AnalyteGroupID					Not required.
AnalyteName	X	X	X		Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment for each TIC.
AnalyteNameContext	X	X	X		Report "CAS" as applicable.
AnalyteType	X	X	X		Report "Target" for all target analytes, "Spike" for all target analytes designated as spike analytes for MS/MSD, "Internal_Standard" for internal standards, "Surrogate" for DMCs, or "TIC" for all TICs.
BiasErrorRatio					Not required.
CASRegistryNumber	X	X	X		Report the CAS Number as it appears in the SOW, and for TICs if known.

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB NCS	Instructions
ClientAnalyteID	X	X	X	Report the CAS Number. For TICs with no CAS Number, report TIC name or as "Unknown-01", then increment with each TIC.
ClientAnalyteName	X	X	X	Report the analytes as they appear in the SOW or as identified for TICs. Report unknown TICs as "Unknown-01", then increment with each TIC.
Comment				Not required.
Counts				Not required.
CountsUncertainty				Not required.
CountsUncertaintyConfidenceLevel				Not required.
CountsUncertaintyDetermination				Not required.
CountsUncertaintyIntervalType				Not required.
CountsUncertaintyLimitHigh				Not required.
CountsUncertaintyLimitLow				Not required.
CountsUncertaintyType				Not required.
CountsUnits				Not required.
DetectionLimit	X	X	X	Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	Report "MDL".
DetectionLimitUnits	X	X	X	Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio				Not required.
Efficiency				Not required.
ExpectedResult				Not required.
ExpectedResultUncertainty				Not required.
ExpectedResultUncertaintyConfidenceLevel				Not required.
ExpectedResultUncertaintyDetermination				Not required.
ExpectedResultUncertaintyIntervalType				Not required.
ExpectedResultUncertaintyLimitHigh				Not required.
ExpectedResultUncertaintyLimitLow				Not required.
ExpectedResultUncertaintyType				Not required.
ExpectedResultUncertaintyUnits				Not required.
ExpectedResultUnits				Not required.
Inclusion				Not required.
LabAnalyteID				Not required.
LabQualifiers	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard (DMCs, internal standards, and spiking analytes only).

TABLE 3. GAS CHROMATOGRAPHY/MASS SPECTROMETRY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	MB/LEB/SB/IB	NCS	Instructions
PeakID	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak. For unknown TICs, report the unique identifiers as applicable. For alkanes, report "Total alkanes" as the identifier.
PercentRecovery					Not required.
PercentRecoveryLimitHigh					Not required.
PercentRecoveryLimitLow					Not required.
PercentRecoveryLimitType					Not required.
PercentRecoveryType					Not required.
QuantitationLimit	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Result	X	X	X		Report the final calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
StandardSource	X	X	X		Report the vendor/manufacturer for this standard.
Wavelength					Not required.
WavelengthUnits					Not required.
AnalyteGroup					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	Report "Pest" or "Aroclor" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
SamplePlusMethod	X	X	X	X	X	
Bottles						Not required.
BottleType						Not required.
ClientID	X	X				Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory						Not required.
ClientMethodCode	X	X	X	X		Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID	X	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	Report "GCECD_External_Standard".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName						Not required.
ClientSampleID	X	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X				Report the date and time the sample was collected.
CollectedEndDate						Not required.
Comment						Not required.
Composite						Not required.
CoolerID						Not required.
CustodyID	X	X				Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch						Not required.
Filtered						Not required.
LabContract	X	X	X	X		Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X				Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
LocationID						Not required.
LocationName						Not required.
MatrixID	X	X	X	X	X	Report "Water" or "Soil" as applicable.
MatrixMedium	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch						Not required.
MethodCategory						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X				Report "Low".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	Report "GC".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X				Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID						Not required.
PhaseAnalyzed						Not required.
Preservative	X	X				Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X		Report the Case Number.
ProjectName						Not required.
QCCategory		X	X	X		Report "Blank" for MB, LEB, IB, or CB; "Spike" for MS; "Spike_Duplicate" for MSD; or "Blank_Spike" for LCS.
QCLinkage		X	X	X		Report "LabReportingBatch" for MS/MSD; "PreparationBatch" for MB and LCS; "AnalysisBatch" for IB; "CleanupBatch" for CB; or "HandlingBatch" for LEB.
QCType	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Cleanup_Blank" for CB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; "Laboratory_Control_Sample" for LCS; or "Non_Client_Sample".
Quarantine	X					Report "Yes" or "No" based on sampling information.
SamplingBatch						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ShippingBatch						Not required.
SiteID						Not required.
SiteName						Not required.
StorageBatch						Not required.
InstrumentQC						Not required.
Characteristic	X	X	X	X		
CharacteristicType	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".
CharacteristicValue	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X		Report "C" for "Temperature".
Comment						Not required.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.
LabZipCode	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisBatch	X	X	X	X	X	Links this analysis to the beginning of a 12-hour period. Report the Lab File ID of the standard (IB for CCV; IB or resolution check for ICAL) that starts the sequence. For the standard at the beginning of a 12-hour period, report the Lab File ID of the standard itself.
AnalysisBatchEnd	X	X	X	X	X	Links this analysis to the QC immediately following a 12-hour period. Report the Lab File ID of the CCV used to close out the 12-hour period.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID						Not required.
AnalysisType	X	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.
Analyst	X	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X	X		Report the volume of final extract added to the sample vial in microliters to at least two significant figures.
AnalyzedAmountUnits	X	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection						Not required.
BackgroundRawData						Not required.
BackgroundType						Not required.
BottleID						Not required.
ClientAnalysisID	X	X	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	X		Report the GC column used.
ColumnInternalDiameter	X	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	X		Report "mm".
ColumnLength	X	X	X	X		Report the Column Length in meters.
ColumnLengthUnits	X	X	X	X		Report "m".
Comment						Not required.
ConfirmationAnalysisID	X	X	X	X		Links an analysis to a confirmation analysis. Report the Lab File ID of the confirmation analysis.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectorID						Not required.
DetectorType	X	X	X	X		Report "ECD".
DilutionFactor	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.
InjectionVolume	X	X	X	X		Report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	X		Report "uL".
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection						Not required.
LabAnalysisID	X	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	X	Report the Lab File ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalLabAnalysisID	X	X	X	X		If a dilution or reinjection is prepared from a previously analyzed sample, report the Lab File ID of the original sample that the dilution or reinjection is prepared from.
PreparationBatch						Not required.
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis	X	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
RunBatch	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard that started the ICAL sequence.
SampleAmount						Not required.
SampleAmountUnits						Not required.
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.
AnalysisGroup						Not required.
Handling	X	X		X		
Analyst						Not required.
BottleID						Not required.
ClientMethodCode						Not required.
ClientMethodID	X	X		X		Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X		X		Report "EPA_CLP".

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientMethodVersion	X	X		X		Report the month and year the SOW was issued.
Comment						Not required.
HandledDate	X	X		X		Enter the date and time TCLP or SPLP extraction began or decanting was performed.
HandlingBatch	X	X		X		Links all samples that were TCLP or SPLP extracted together or decanted together. Report a unique identifier for each batch.
HandlingType	X	X		X		Report "TCLP" or "SPLP" for extractions. Report "Decanted" if water was decanted from soil/sediment samples; otherwise report "Not_decanted".
InitialAmount						Not required.
InitialAmountUnits						Not required.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X		X		Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X		X		Report "EPA_CLP".
MethodVersion	X	X		X		Report the month and year the SOW was issued.
ProcedureID						Not required.
ProcedureName						Not required.
SampleAmount						Not required.
SampleAmountUnits						Not required.
ReportedResult	X	X	X	X		
AnalysisGroupID						Not required.
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X		Report "Target" for all target analytes or "Spike" for all target analytes designated as spike analytes for MS/MSD and LCS analysis.
BiasErrorRatio						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Number as it appears in the SOW.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientAnalyteID	X	X	X	X		Report CAS Number.
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit						Not required.
ClientDetectionLimitUnits						Not required.
ClientQuantitationLimit	X	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment						Not required.
DetectionLimit	X	X	X	X		For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio						Not required.
ExpectedResult						Not required.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits						Not required.
LabAnalysisID	X	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X				Report "Preliminary" or "Final" as applicable.
PeakID						Not required.
PercentDifference	X	X	X	X		For Confirmation analyses, report the Percent Difference between the reported results and the confirmation result to the nearest whole percent (excluding IB).
PercentDifferenceLimitHigh	X	X	X	X		Report the upper limit for the Percent Difference to the nearest whole percent (excluding IB).
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType	X	X	X	X		Report "Method" (excluding IB).

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability					Instructions
	Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
QuantitationLimit	X	X	X	X		For target analytes, report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime						Not required.
RetentionTimeUnits						Not required.
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.
RPDType						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
PreparationPlusCleanup	X	X	X	X		
AliquotAmount	X	X	X	X		Report the sample amount in grams for soil/sediment or milliliters for aqueous/water to at least three significant figures.
AliquotAmountUnits	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X	X		Report the Analyst's initials.
BottleID						Not required.
CleanedUpDate	X	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X	X		Report "GPC", "Florisil", "Sulfur", or "Sulfuric_Acid" as applicable.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X		Report the month and year the SOW was issued.
Comment						Not required.
Efficiency						Not required.
FinalAmount	X	X	X	X		Report the Final Amount of material produced upon completion of this prep or cleanup in microliters.
FinalAmountUnits	X	X	X	X		Report "uL".
InitialAmount	X	X	X	X		Report the initial amount of extracted sample used for this cleanup method in microliters.
InitialAmountUnits	X	X	X	X		Report "uL".
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
LotNumber	X	X	X	X		Report the manufacturer's lot number for the Florisil cartridges used.
MethodCode						Not required.
MethodID	X	X	X	X		Report "SOM02.4".
MethodModificationDescription						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X	X		Links all samples that were prepared together. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Sep_Funnel", "Liq_Liq", or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution.
PreparedDate	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.
SampleAmount						Not required.
SampleAmountUnits						Not required.
Solvent						Not required.
Analyte	X	X	X	X		
AmountAdded	X	X	X	X		Volume of surrogate standard or spiking solution added in microliters.
AmountAddedUnits	X	X	X	X		Report "uL".
AmountAddedLocation	X	X	X	X		For sample, MB, CB, or MS/MSD, report "Aliquot"; for LCS or IB, report "Standard".
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X		Report "Target" for all target analytes; "Spike" for all target analytes designated as spike analytes for MS/MSD or LCS analysis; or "Surrogate" for surrogate compounds.
BiasErrorRatio						Not required.
CalibrationBasis						Not required.
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X		Report CAS Number.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectionLimit	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult	X	X	X	X		Report the theoretical final calculated concentration for MS/MSD and LCS. Report surrogates in nanograms.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ExpectedResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment and "ug/L" for aqueous/water (or "mg/L" for TCLP). Report "ng" for surrogates.
Inclusion						Not required.
IntermediateResult	X	X	X	X		Report the on-column amount unadjusted for sample weight/volume, percent solids, or dilution factor, in nanograms, from the raw data. Leave blank if undetected.
IntermediateResultLimitHigh						Not required.
IntermediateResultLimitLow						Not required.
IntermediateResultLimitType						Not required.
IntermediateResultUnits	X	X	X	X		Report "ng".
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard.
Mass						Not required.
MassLimitHigh						Not required.
MassLimitLow						Not required.
MassLimitType						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown						Not required.
PercentBreakdownLimitHigh						Not required.
PercentBreakdownLimitType						Not required.
PercentDifference						Not required.
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentMatch						Not required.
PercentRecovery	X	X	X	X		Report the final calculated Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitHigh	X	X	X	X		Report the upper limit for the Percent Recovery of the spikes and surrogates to the nearest whole percent.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
PercentRecoveryLimitLow	X	X	X	X		Report the lower limit for the Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitType	X	X	X	X		Report "Method".
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationBasis						Not required.
QuantitationLimit	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Response						Not required.
ResponseLimitHigh						Not required.
ResponseLimitLow						Not required.
ResponseLimitType						Not required.
ResponseUnits						Not required.
Result	X	X	X	X		Report the calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RPD		X				Report the RPD to the nearest percent.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
RPDLimitHigh		X				Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType		X				Report "Method".
RPDType						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardConcentration	X	X	X	X		Report the concentration of the surrogate standard or spiking solution used in ug/L.
StandardConcentrationUnits						Report "ug/L".
StandardDeviation						Not required.
StandardDeviationUnits						Not required.
StandardFinalAmount						Not required.
StandardFinalAmountUnits						Not required.
StandardID						Not required.
StandardSource	X	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
AnalyteComparison						Not required.
AnalyteGroup						Not required.
Peak	X	X	X	X		
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
DetectionLimit						Not required.
DetectionLimitType						Not required.
DetectionLimitUnits						Not required.
DifferenceErrorRatio						Not required.
Efficiency						Not required.
Inclusion						Not required.
IntermediateResult	X	X	X	X		Report the on-column amount in nanograms from the raw data. Leave blank if compound is not detected.
IntermediateResultLimitHigh						Not required.
IntermediateResultLimitLow						Not required.
IntermediateResultLimitType						Not required.
IntermediateResultUnits	X	X	X	X		Report "ng".
LabQualifiers						Not required.
ManualIntegration	X	X	X	X		Report "Yes" if this peak was manually integrated; otherwise report "No".
Mass						Not required.
MassLimitHigh						Not required.
MassLimitLow						Not required.
MassLimitType						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRetentionTime						Not required.
MeanRetentionTimeLimitHigh						Not required.
MeanRetentionTimeLimitLow						Not required.
MeanRetentionTimeLimitType						Not required.
MeanRetentionTimeUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X		Report the peak identifier as used by the laboratory to uniquely identify this peak.
PeakRatio						Not required.
PeakRatioLimitHigh						Not required.
PeakRatioLimitLow						Not required.
PeakRatioLimitType						Not required.
PercentDifference						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentRatio						Not required.
PercentRatioLimitHigh						Not required.
PercentRatioLimitLow						Not required.
PercentRatioLimitType						Not required.
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationLimit						Not required.
QuantitationLimitType						Not required.
QuantitationLimitUnits						Not required.
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Resolution						Not required.
ResolutionLimitHigh						Not required.
ResolutionLimitLow						Not required.
ResolutionLimitType						Not required.
ResolutionType						Not required.
ResolutionUnits						Not required.
Response	X	X	X	X		Report the actual peak area or peak height from the raw data.
ResponseLimitHigh						Not required.
ResponseLimitLow						Not required.
ResponseLimitType						Not required.
ResponseType						Not required.
ResponseUnits	X	X	X	X		Report "Peak_Area" or "Peak_Height".
Result						Not required.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ResultType						Not required.
ResultUncertainty						Not required.
ResultUnits						Not required.
RetentionTime	X	X	X	X		Report the actual retention time in decimal minutes from the raw data for this peak.
RetentionTimeLimitLow	X	X	X	X		Report the lower limit for this retention time in decimal minutes for the internal standards.
RetentionTimeLimitType	X	X	X	X		Report "Method".
RetentionTimeUnits	X	X	X	X		Report "Minutes".
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardDeviation						Not required.
StandardDeviationUnits						Not required.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
PeakComparison						Not required.
PeakReplicate						Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
Header	X	X	X	X	
ClientID	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName					Not required.
Comment					Not required.
DateFormat	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabDataPackageID	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	Report "Pest" or "Aroclor" as applicable.
LabDataPackageVersion	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabNarrative					Not required.
LabQualifiersDefinition	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	Report the Case Number.
ProjectName					Not required.
SiteID					Not required.
SiteName					Not required.
SamplePlusMethod					Not required.
InstrumentQC	X	X	X	X	
ClientInstrumentQCType	X	X			For pesticides, for RESC and standards, report "1" if using a single mixture to calibrate instrument. Report "2" if using two mixtures to calibrate instrument.
ClientMethodCode	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X		Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID	X	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Comment					Not required.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	X	Report the EPA Sample Number. For ICAL, report the EPA Sample Number of the first standard.
LabMethodID					Not required.
LabMethodName					Not required.
LabName	X	X	X	X	Report the Lab Name.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	X	Report "RunBatch" for ICAL and IPC; "AnalysisBatch" for CCV; or "CleanupBatch" for FLO and GPC.
QCType	X	X	X	X	Report "Instrument_Performance_Check_Tune" for RESC; "Instrument_Performance_Check_PEM" for the PEM standards that are part of the ICAL; "Initial_Calibration" for calibration; "Continuing_Calibration_Verification" for CCV; "Florisil_Cartridge_Check" for the Florisil cartridge check; or "GPC_Calibration_Check" for the GPC calibration check.
ContactInformation	X	X	X	X	
LabAddress1	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType					Not required.
LabState	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
LabZipCode	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
AnalysisBatch			X		Links this analysis to the beginning of a 12-hour period. Report the Lab File ID of the standard (IB for CCV; IB or resolution check for ICAL) that starts this sequence. For the standard that starts the 12-hour period, enter the Lab File ID of the standard itself.
AnalysisBatchEnd			X		Links this analysis to the end of a 12-hour period. Report the Lab File ID of the CCV that ends this sequence. For the closing CCV that closes the 12-hour period, report the Lab File ID of the standard itself.
AnalysisDuration					Not required.
AnalysisDurationUnits					Not required.
AnalysisGroupID		X			Links a group of analyses together that are used for the multipoint initial calibration. Report the Lab File ID of the standard that starts this ICAL sequence.
AnalysisType	X	X	X	X	For IPC, FLO, and GPC report "Initial". For ICAL/CCV, report the calibration level used.
Analyst	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount	X	X	X	X	Report the volume of standard places on the instrument for analysis in microliters.
AnalyzedAmountUnits	X	X	X	X	Report "uL".
AnalyzedDate	X	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection					Not required.
BackgroundRawData					Not required.
BackgroundType					Not required.
BottleID					Not required.
ClientAnalysisID	X	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode					Not required.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	X	Report the GC Column used.
ColumnInternalDiameter	X	X	X	X	Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	X	Report "mm".

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
ColumnLength	X	X	X	X	Report the GC Column Length in meters.
ColumnLengthUnits	X	X	X	X	Report "m".
Comment					Not required.
ConfirmationAnalysisID					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectorID					Not required.
DetectorType	X	X	X	X	Report "ECD".
DilutionFactor	X	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency					Not required.
HeatedPurge					Not required.
Inclusion		X			Report "Yes" if the ICAL standard is to be included in the calibration curve; otherwise report "No".
InjectionVolume	X	X	X	X	Report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	X	Report "uL" as applicable.
InstrumentID	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection					Not required.
LabAnalysisID	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	Report the Lab File ID.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report month and year the SOW was issued.
OriginalLabAnalysisID					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
PreparationBatch					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
ReferenceDate					Not required.
ResultBasis					Not required.
RunBatch	X	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard that started the ICAL sequence.
SampleAmount					Not required.
SampleAmountUnits					Not required.
Temperature					Not required.
TemperatureUnits					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
Yield					Not required.
AnalysisGroup		X			
AnalysisGroupID		X			This links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard that starts this calibration sequence.
AnalysisType		X			Report "Initial_Calibration".
Comment					Not required.
Handling					Not required.
ReportedResult					Not required.
PreparationPlusCleanup				X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
Analyst				X	Report the Analyst's initials.
BottleID					Not required.
CleanedUpDate				X	Report the date and time the sample was cleaned up.
CleanupBatch				X	Links all samples that were cleaned up together. Report the Lab File ID of the associated cleanup blank.
CleanupType				X	Report "GPC" or "Florisil" as applicable.
ClientMethodCode					Not required.
ClientMethodID				X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource				X	Report "EPA_CLP".
ClientMethodVersion				X	Report the month and year the SOW was issued.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
Comment					Not required.
Efficiency					Not required.
FinalAmount				X	Report the final amount of material produced upon completion of this prep or cleanup in microliters.
FinalAmountUnits				X	Report "uL".
InitialAmount				X	Report the initial amount of extracted sample used for this cleanup method in microliters.
InitialAmountUnits				X	Report "uL".
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
LotNumber				X	Report the manufacturer's lot number for the Florisil cartridges used.
MethodCode					Not required.
MethodID				X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource				X	Report "EPA_CLP".
MethodVersion				X	Report the month and year the SOW was issued.
PreparationBatch					Not required.
PreparationPlusCleanupType				X	Report "Cleanup".
PreparationType					Not required.
PreparedDate					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
SampleAmount					Not required.
SampleAmountUnits					Not required.
Solvent					Not required.
Analyte	X	X	X	X	
AmountAdded	X	X	X	X	Volume of surrogate or spiking standard added in microliters.
AmountAddedUnits	X	X	X	X	Report "uL".
AmountAddedLocation	X	X	X	X	Report "Standard".
AnalyteGroupID					Not required.
AnalyteName	X	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	Report "CAS".
AnalyteType	X	X	X	X	Report "Target" for all target analytes or "Surrogate" for surrogate compounds.
BiasErrorRatio					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
CalibrationBasis		X			Report "Peak" under the AnalysisGroup node.
CalibrationFactor					Not required.
CalibrationFactorUnits					Not required.
CalibrationType					Not required.
CASRegistryNumber	X	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	Report CAS Number.
ClientAnalyteName	X	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectionLimit					Not required.
DetectionLimitType					Not required.
DetectionLimitUnits					Not required.
DifferenceErrorRatio					Not required.
Efficiency					Not required.
ExpectedResult	X	X	X	X	Report the final amount for all applicable target analytes and surrogates.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits					Not required.
ExpectedResultUnits	X	X	X	X	Report "ng".
Inclusion		X			Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult	X	X	X	X	Report the on-column amount unadjusted for sample weight/volume, percent solids, or dilution factor, in nanograms, from the raw data.
IntermediateResultLimitHigh					Not required.
IntermediateResultLimitLow					Not required.
IntermediateResultLimitType					Not required.
IntermediateResultUnits	X	X	X	X	Report "ng".
LabAnalyteID					Not required.
LabQualifiers	X	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass					Not required.
MassLimitHigh					Not required.
MassLimitLow					Not required.
MassLimitType					Not required.
MassUnits					Not required.
MeanCalibrationFactor					Not required.
MeanCalibrationFactorUnits					Not required.
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown	X				For pesticides, report the calculated percent breakdown for 4,4'-DDT and Endrin to the nearest whole percent.
PercentBreakdownLimitHigh	X				Report the upper limit for the percent breakdown to the nearest whole percent.
PercentBreakdownLimitType	X				Report "Method".
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentMatch					Not required.
PercentRecovery				X	Report the final calculated Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh				X	Report the upper limit for the Percent Recovery to the nearest whole percent.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
PercentRecoveryLimitLow				X	Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType				X	Report "Method".
PercentRecoveryType					Not required.
PercentRSD					Not required.
PercentRSDLimitHigh					Not required.
PercentRSDLimitLow					Not required.
PercentRSDLimitType					Not required.
QuantitationBasis		X			Report "External_Standard" under the AnalysisGroup node.
QuantitationLimit					Not required.
QuantitationLimitType					Not required.
QuantitationLimitUnits					Not required.
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Response					Not required.
ResponseLimitHigh					Not required.
ResponseLimitLow					Not required.
ResponseLimitType					Not required.
ResponseUnits					Not required.
Result					Not required.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType					Not required.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits					Not required.
RPD					Not required.
RPDLimitHigh					Not required.
RPDLimitType					Not required.
RPDType					Not required.
RRF					Not required.
RRFLimitLow					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
RRFLimitType					Not required.
StandardConcentration	X	X	X	X	Report the concentration of standard used in micrograms per liter.
StandardConcentrationUnits	X	X	X	X	Report "ug/L".
StandardDeviation					Not required.
StandardDeviationUnits					Not required.
StandardFinalAmount					Not required.
StandardFinalAmountUnits					Not required.
StandardID	X	X	X	X	Report the laboratory-assigned identifier for this standard.
StandardSource	X	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.
AnalyteComparison					Not required.
AnalyteGroup					Not required.
Peak	X	X	X	X	
CalibrationFactor		X	X		Report the calculated Calibration Factor.
CalibrationFactorUnits		X	X		Report the units for the Calibration Factor.
CalibrationType		X	X		Report "Calibration_Factor" under the AnalysisGroup node.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
DetectionLimit					Not required.
DetectionLimitType					Not required.
DetectionLimitUnits					Not required.
DifferenceErrorRatio					Not required.
Efficiency					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
Inclusion		X			Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult		X	X		Report the on-column amount in nanograms from the raw data.
IntermediateResultLimitHigh					Not required.
IntermediateResultLimitLow					Not required.
IntermediateResultLimitType					Not required.
IntermediateResultUnits		X	X		Report "ng".
LabQualifiers					Not required.
ManualIntegration	X	X	X	X	Report "Yes" if this peak was manually integrated; otherwise report "No".
Mass					Not required.
MassLimitHigh					Not required.
MassLimitLow					Not required.
MassLimitType					Not required.
MassUnits					Not required.
MeanCalibrationFactor		X			Report the calculated Mean Calibration Factor under the AnalysisGroup node only.
MeanCalibrationFactorUnits		X			Report the units for the Mean Calibration Factor under the AnalysisGroup node only.
MeanRetentionTime		X			Report the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitHigh		X			Report the upper limit for the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitLow		X			Report the lower limit for the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitType		X			Report "Method".
MeanRetentionTimeUnits		X			Report "Minutes".
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X	X	Report the peak identifier as used by the laboratory to uniquely identify this peak. This identifier must be consistent throughout an analytical sequence.
PeakRatio					Not required.
PeakRatioLimitHigh					Not required.
PeakRatioLimitLow					Not required.
PeakRatioLimitType					Not required.
PercentDifference			X		Report the calculated Percent Difference for this peak to the nearest tenth of a percent.
PercentDifferenceLimitHigh			X		Report the upper limit for the Percent Difference for this peak to the nearest tenth of a percent.
PercentDifferenceLimitLow			X		Report the lower limit for the Percent Difference for this peak to the nearest tenth of a percent.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
PercentDifferenceLimitType			X		Report "Method".
PercentRatio					Not required.
PercentRatioLimitHigh					Not required.
PercentRatioLimitLow					Not required.
PercentRatioLimitType					Not required.
PercentRecovery					Not required.
PercentRecoveryLimitHigh					Not required.
PercentRecoveryLimitLow					Not required.
PercentRecoveryLimitType					Not required.
PercentRecoveryType					Not required.
PercentRSD		X			Report the calculated %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitHigh		X			Report the upper limit for the %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitLow					Not required.
PercentRSDLimitType		X			Report "Method".
QuantitationLimit					Not required.
QuantitationLimitType					Not required.
QuantitationLimitUnits					Not required.
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Resolution	X	X	X		For pesticides, report the percent resolution for midpoint INDA, INDB, or INDC initial calibration standards only. Report resolutions for all PEMs used in the initial and calibration verification standards.
ResolutionLimitHigh					Not required.
ResolutionLimitLow	X	X	X		Report the lower limit for the percent resolution.
ResolutionLimitType	X	X	X		Report "Method".
ResolutionType	X	X	X		Report "Percent_Resolution".
ResolutionUnits	X	X	X		Report "Percent".
Response	X	X	X	X	Report the actual Peak Area (or Peak Height) from the raw data.
ResponseLimitHigh					Not required.
ResponseLimitLow					Not required.
ResponseLimitType					Not required.
ResponseType					Not required.
ResponseUnits	X	X	X	X	Report "Peak_Area" or "Peak_Height".
Result					Not required.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.

TABLE 4. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
ResultLimitType					Not required.
ResultType					Not required.
ResultUncertainty					Not required.
ResultUnits					Not required.
RetentionTime	X	X	X	X	Report the actual retention time in decimal minutes from the raw data for this peak.
RetentionTimeLimitHigh	X	X	X	X	Report the upper limit for this retention time in decimal minutes.
RetentionTimeLimitLow	X	X	X	X	Report the lower limit for this retention time in decimal minutes.
RetentionTimeLimitType	X	X	X	X	Report "Method".
RetentionTimeUnits	X	X	X	X	Report "Minutes".
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
StandardDeviation					Not required.
StandardDeviationUnits					Not required.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.
PeakComparison					Not required.
PeakReplicate					Not required.

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TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability					Instructions
	Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	Report "Pest" or "Aroclor" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability					Instructions
	Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	
SamplePlusMethod	X	X	X	X	X	
ClientID	X	X				Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory						Not required.
ClientMethodCode	X	X	X	X		Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID	X	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	Report "GCECD_External_Standard".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName						Not required.
ClientSampleID	X	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X				Report the date and time the sample was collected.
CollectedEndDate						Not required.
Comment						Not required.
Composite						Not required.
CoolerID						Not required.
CustodyID	X	X				Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch						Not required.
Filtered						Not required.
LabContract	X	X	X	X		Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X				Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID						Not required.
LocationName						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
MatrixID	X	X	X	X	X	Report "Water" or "Soil " as applicable.
MatrixMedium	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch						Not required.
MethodCategory						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X				Report "Low".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	Report "GC".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X				Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID						Not required.
PhaseAnalyzed						Not required.
Preservative	X	X				Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X		Report the Case Number.
ProjectName						Not required.
QCCategory		X	X	X		Report "Blank" for MB, LEB, IB, or CB; "Spike" for MS; "Spike_Duplicate" for MSD; or "Blank_Spike" for LCS.
QCLinkage		X	X	X		Report "LabReportingBatch" for MS/MSD; "PreparationBatch" for MB and LCS; AnalysisBatch for IB; or "CleanupBatch" for CB.
QCType	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Cleanup_Blank" for CB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; "Laboratory_Control_Sample" for LCS; or "Non_Client_Sample".
Quarantine	X					Report "Yes" or "No" based on sampling information.
SamplingBatch						Not required.
ShippingBatch						Not required.
SiteID						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
SiteName						Not required.
StorageBatch						Not required.
InstrumentQC						Not required.
Characteristic	X	X	X	X		
CharacteristicType	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".
CharacteristicValue	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X		Report "C" for "Temperature".
Comment						Not required.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
LabZipCode	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisBatch	X	X	X	X	X	Links this analysis to the beginning of the 12-hour period. Report the Lab File ID of the standard (IB for CCV; IB or resolution check for ICAL) that starts the sequence. For the standard at the beginning of the 12-hour period, report the Lab File ID of the standard itself.
AnalysisBatchEnd	X	X	X	X	X	Links this analysis to the QC immediately following a 12-hour period. Report the Lab File ID of the CCV used to close out the 12-hour period.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID						Not required.
AnalysisType	X	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.
Analyst	X	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X	X		Report the volume of final extract added to the sample vial in microliters to at least two significant figures.
AnalyzedAmountUnits	X	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X	X		Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	X		Report the GC Column used.
ColumnInternalDiameter	X	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	X		Report "mm".
ColumnLength	X	X	X	X		Report the Column Length in meters.
ColumnLengthUnits	X	X	X	X		Report "m".

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
Comment						Not required.
ConfirmationAnalysisID	X	X	X	X		Links an analysis to a confirmation analysis. Report the Lab File ID of the confirmation analysis.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectorID						Not required.
DetectorType	X	X	X	X		Report "ECD".
DilutionFactor	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.
InjectionVolume	X	X	X	X		Report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	X		Report "uL".
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	X	Report the Lab File ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis	X	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
RunBatch	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard that started the ICAL sequence.
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.
AnalysisGroup						Not required.
Handling						Not required.
ReportedResult	X	X	X	X		
AnalysisGroupID						Not required.
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X		Report "Target" for all target analytes or "Spike" for all target analytes designated as spike analytes for MS/MSD and LCS analysis.
BiasErrorRatio						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Numbers as they appear in the SOW.
ClientAnalyteID	X	X	X	X		Report CAS Number.
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit						Not required.
ClientDetectionLimitUnits						Not required.
ClientQuantitationLimit	X	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment						Not required.
DetectionLimit	X	X	X	X		For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
DetectionLimitType	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio						Not required.
ExpectedResult						Not required.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits						Not required.
LabAnalysisID	X	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X				Report "Preliminary" or "Final" as applicable.
PeakID						Not required.
PercentDifference	X	X	X	X		For Confirmation analyses, report the Percent Difference between the reported results and the confirmation result to the nearest whole percent (excluding IB).
PercentDifferenceLimitHigh	X	X	X	X		Report the upper limit for the Percent Difference to the nearest whole percent (excluding IB).
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
QuantitationLimit	X	X	X	X		For target analytes, report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime						Not required.
RetentionTimeUnits						Not required.
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.
RPDType						Not required.
PreparationPlusCleanup	X	X	X	X		
AliquotAmount	X	X	X	X		Report the sample amount in grams for soil/sediment or milliliters for aqueous/water to at least three significant figures.
AliquotAmountUnits	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X	X		Report the Analyst's initials.
CleanedUpDate	X	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X	X		Report "GPC", "Florisil", "Sulfur", or "Sulfuric_Acid" as applicable.
ClientMethodCode						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientMethodID	X	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X		Report the month and year the SOW was issued.
Comment						Not required.
FinalAmount	X	X	X	X		Report the Final Amount of material produced upon completion of this prep or cleanup in microliters.
FinalAmountUnits	X	X	X	X		Report "uL".
InitialAmount	X	X	X	X		Report the initial amount of extracted sample used for this cleanup method in microliters.
InitialAmountUnits	X	X	X	X		Report "uL".
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
LotNumber						Not required.
MethodCode						Not required.
MethodID	X	X	X	X		Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X	X		Links all samples that were prepared together. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Sep_Funnel", "Liq_Liq", or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution.
PreparedDate	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
Solvent						Not required.
Analyte	X	X	X	X		
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X		Report "Target" for all target analytes; "Spike" for all target analytes designated as spike analytes for MS/MSD or LCS; or "Surrogate" for surrogate compounds.
BiasErrorRatio						Not required.
CalibrationBasis						Not required.
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X		Report CAS Number.
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
DetectionLimit	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult	X	X	X	X		Report the theoretical final calculated concentration for MS/MSD and LCS. Report surrogates in nanograms.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Inclusion						Not required.
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report the qualifiers as specified in the SOW.
LotNumber	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard.
Mass						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown						Not required.
PercentBreakdownLimitHigh						Not required.
PercentBreakdownLimitType						Not required.
PercentDifference						Not required.
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
PercentRecovery	X	X	X	X		Report the final calculated Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitHigh	X	X	X	X		Report the upper limit for the Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitLow	X	X	X	X		Report the lower limit of the Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitType	X	X	X	X		Report "Method".
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationBasis						Not required.
QuantitationLimit	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result	X	X	X	X		Report the final calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RPD		X				Report the RPD to the nearest percent.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
RPDLimitHigh		X				Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType		X				Report "Method".
RPDType						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardSource	X	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
AnalyteGroup						Not required.
Peak						Not required.
PeakComparison						Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
Header	X	X	X	X	
ClientID	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName					Not required.
Comment					Not required.
DateFormat	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	Report the Contract Number.
LabContractModificationDescription					Not required.
LabContractModificationID					Not required.
LabDataPackageID	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	Report "Pest" or "Aroclor" as applicable.
LabDataPackageVersion	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabNarrative					Not required.
LabQualifiersDefinition	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	Report the Case Number.
ProjectName					Not required.
SiteID					Not required.
SiteName					Not required.
SamplePlusMethod					Not required.
InstrumentQC	X	X	X	X	
ClientInstrumentQCType	X	X			For Pesticides, for RESC and standards, report "1" if using a single mixture to calibrate instrument. Report "2" if using two mixtures to calibrate instrument.
ClientMethodCode	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X		X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID	X	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Comment					Not required.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	X	Report the EPA Sample Number. For ICAL, report the EPA Sample Number of the first standard.
LabMethodID					Not required.
LabMethodName					Not required.
LabName	X	X	X	X	Report the Lab Name.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	X	Report "RunBatch" for ICAL and IPC; "AnalysisBatch" for CCV; or "CleanupBatch" for FLO and GPC.
QCType	X	X	X	X	Report "Instrument_Performance_Check_Tune" for RESC; "Instrument_Performance_Check_PEM" for the PEM standards that are part of the ICAL; "Initial_Calibration" for calibration; "Continuing_Calibration_Verification" for CCV; "Florisil_Cartridge_Check" for the Florisil cartridge check; or "GPC_Calibration_Check" for the GPC calibration check.
ContactInformation	X	X	X	X	
LabAddress1	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType					Not required.
LabState	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
LabZipCode	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
AnalysisBatch			X		Links this analysis to the beginning of a 12-hour period. Report the Lab File ID of the standard (IB for CCV; IB or resolution check for ICAL) that starts this sequence. For the standard that starts the 12-hour period, enter the Lab File ID of the standard itself.
AnalysisBatchEnd			X		Links this analysis to the end of a 12-hour period. Report the Lab File ID of the CCV that ends this sequence. For the closing CCV that closes the 12-hour period, report the Lab File ID of the standard itself.
AnalysisDuration					Not required.
AnalysisDurationUnits					Not required.
AnalysisGroupID		X			Links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard that starts this ICAL sequence.
AnalysisType	X	X	X	X	For IPC, FLO, and GPC, report "Initial". For ICAL/CCV, report the calibration level used.
Analyst	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount					Not required.
AnalyzedAmountUnits					Not required.
AnalyzedDate	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X	X	Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode					Not required.
ClientMethodID	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required.
ClientMethodName					Not required.
ClientMethodSource	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	Report the month and year the SOW was issued.
Column	X	X	X	X	Report the GC Column used.
ColumnInternalDiameter	X	X	X	X	Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	X	Report "mm".
ColumnLength	X	X	X	X	Report the GC Column Length in meters.
ColumnLengthUnits	X	X	X	X	Report "m".
Comment					Not required.
ConfirmationAnalysisID					Not required.
Counts					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectorID					Not required.
DetectorType	X	X	X	X	Report "ECD".
DilutionFactor	X	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency					Not required.
HeatedPurge					Not required.
Inclusion		X			Report "Yes" if the ICAL standard is to be included in the calibration curve; otherwise report "No".
InjectionVolume	X	X	X	X	Report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	X	Report "uL".
InstrumentID	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	Report the Lab File ID.
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.
LabName					Not required.
MethodCode					Not required.
MethodID	X	X	X	X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
ReferenceDate					Not required.
ResultBasis					Not required.
RunBatch	X	X	X	X	Links this analysis to an initial calibration. Report the Lab File ID of the standard that started the ICAL sequence.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
Temperature					Not required.
TemperatureUnits					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
Yield					Not required.
AnalysisGroup		X			
AnalysisGroupID		X			This links a group of analyses together that are used for the initial calibration. Report the Lab File ID of the standard that starts this calibration sequence.
AnalysisType		X			Report "Initial_Calibration".
Comment					Not required.
Handling					Not required.
ReportedResult					Not required.
PreparationPlusCleanup				X	
AliquotAmount					Not required.
AliquotAmountUnits					Not required.
Analyst				X	Report the Analyst's initials.
CleanedUpDate				X	Report the date and time the sample was cleaned up.
CleanupBatch				X	Links all samples that were cleaned up together. Report the Lab File ID of the associated cleanup blank.
CleanupType				X	Report "GPC" or "Florisil" as applicable.
ClientMethodCode					Not required.
ClientMethodID				X	Report "SOM02.4".
ClientMethodModificationDescription					Not required.
ClientMethodModificationID					Not required
ClientMethodName					Not required.
ClientMethodSource				X	Report "EPA_CLP".
ClientMethodVersion				X	Report the month and year the SOW was issued.
Comment					Not required.
FinalAmount				X	Report the final amount of material produced upon completion of this prep or cleanup step in microliters.
FinalAmountUnits				X	Report "uL".
InitialAmount				X	Report the initial amount of extracted sample used for this cleanup method in microliters.
InitialAmountUnits				X	Report "uL".
LabID					Not required.
LabMethodID					Not required.
LabMethodName					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability				Instructions
	IPC	ICAL	CCV	FLO/GPC	
LabName					Not required.
LotNumber				X	Report the manufacturer's lot number for the Florisil cartridges.
MethodCode					Not required.
MethodID				X	Report "SOM02.4".
MethodModificationDescription					Not required.
MethodModificationID					Not required.
MethodName					Not required.
MethodSource				X	Report "EPA_CLP".
MethodVersion					Report the month and year the SOW was issued.
PreparationBatch					Not required.
PreparationPlusCleanupType					Report "Cleanup".
PreparationType					Not required.
PreparedDate					Not required.
ProcedureID					Not required.
ProcedureName					Not required.
Solvent					Not required.
Analyte	X	X	X	X	
AnalyteGroupID					Not required.
AnalyteName	X	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	Report "CAS".
AnalyteType	X	X	X	X	Report "Target" for all target analytes or "Surrogate" for surrogate standards.
BiasErrorRatio					Not required.
CalibrationBasis		X			Report "Peak" under the AnalysisGroup node.
CalibrationFactor					Not required.
CalibrationFactorUnits					Not required.
CalibrationType					Not required.
CASRegistryNumber	X	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	Report CAS Number.
ClientAnalyteName	X	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
Counts					Not required.
CountsUncertainty					Not required.
CountsUncertaintyConfidenceLevel					Not required.
CountsUncertaintyDetermination					Not required.
CountsUncertaintyIntervalType					Not required.
CountsUncertaintyLimitHigh					Not required.
CountsUncertaintyLimitLow					Not required.
CountsUncertaintyType					Not required.
CountsUnits					Not required.
DetectionLimit					Not required.
DetectionLimitType					Not required.
DetectionLimitUnits					Not required.
DifferenceErrorRatio					Not required.
Efficiency					Not required.
ExpectedResult	X	X	X	X	Report the final amount for all target analytes and surrogates.
ExpectedResultUncertainty					Not required.
ExpectedResultUncertaintyConfidenceLevel					Not required.
ExpectedResultUncertaintyDetermination					Not required.
ExpectedResultUncertaintyIntervalType					Not required.
ExpectedResultUncertaintyLimitHigh					Not required.
ExpectedResultUncertaintyLimitLow					Not required.
ExpectedResultUncertaintyType					Not required.
ExpectedResultUncertaintyUnits					Not required.
ExpectedResultUnits	X	X	X	X	Report "ng".
Inclusion		X			Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabAnalyteID					Not required.
LabQualifiers	X	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass					Not required.
MassUnits					Not required.
MeanCalibrationFactor					Not required.
MeanCalibrationFactorUnits					Not required.
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
PeakID	X	X	X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown	X				For pesticides, report the calculated percent breakdown for 4,4'-DDT and Endrin to the nearest whole percent.
PercentBreakdownLimitHigh	X				Report the upper limit for the percent breakdown to the nearest whole percent.
PercentBreakdownLimitType	X				Report "Method".
PercentDifference					Not required.
PercentDifferenceLimitHigh					Not required.
PercentDifferenceLimitLow					Not required.
PercentDifferenceLimitType					Not required.
PercentRecovery				X	Report the final calculated Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh				X	Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow				X	Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType				X	Report "Method".
PercentRecoveryType					Not required.
PercentRSD					Not required.
PercentRSDLimitHigh					Not required.
PercentRSDLimitLow					Not required.
PercentRSDLimitType					Not required.
QuantitationBasis		X			Report "External_Standard" under the AnalysisGroup node.
QuantitationLimit					Not required.
QuantitationLimitType					Not required.
QuantitationLimitUnits					Not required.
ReportingLimit					Not required.
ReportingLimitType					Not required.
ReportingLimitUnits					Not required.
Result					Not required.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType					Not required.
ResultUncertainty					Not required.
ResultUncertaintyConfidenceLevel					Not required.
ResultUncertaintyDetermination					Not required.
ResultUncertaintyIntervalType					Not required.
ResultUncertaintyLimitHigh					Not required.
ResultUncertaintyLimitLow					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
ResultUncertaintyType					Not required.
ResultUncertaintyUnits					Not required.
ResultUnits					Not required.
RPD					Not required.
RPDLimitHigh					Not required.
RPDLimitType					Not required.
RPDType					Not required.
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
StandardSource	X	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.
AnalyteGroup					Not required.
Peak	X	X	X	X	
CalibrationFactor		X	X		Report the calculated Calibration Factor.
CalibrationFactorUnits		X	X		Report the units for the Calibration Factor.
CalibrationType		X			Report "Calibration_Factor" under the AnalysisGroup node.
Coeffa0					Not required.
Coeffa1					Not required.
Coeffa2					Not required.
Coeffa3					Not required.
CoeffOfDetermination					Not required.
CoeffOfDeterminationLimitLow					Not required.
CoeffOfDeterminationLimitType					Not required.
Comment					Not required.
CorrelationCoeff					Not required.
CorrelationCoeffLimitLow					Not required.
CorrelationCoeffLimitType					Not required.
DifferenceErrorRatio					Not required.
Efficiency					Not required.
Inclusion		X			Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabQualifiers					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
Mass					Not required.
MassLimitHigh					Not required.
MassLimitLow					Not required.
MassLimitType					Not required.
MassUnits					Not required.
MeanCalibrationFactor		X			Report the calculated Mean Calibration Factor under the AnalysisGroup node only.
MeanCalibrationFactorUnits		X			Report the units for the Mean Calibration Factor under the AnalysisGroup node only.
MeanRetentionTime		X			Report the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitHigh		X			Report the upper limit for the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitLow		X			Report the lower limit for the mean retention time in decimal minutes from the ICAL.
MeanRetentionTimeLimitType		X			Report "Method".
MeanRetentionTimeUnits		X			Report "Minutes".
MeanRRF					Not required.
MeanRRFLimitLow					Not required.
MeanRRFLimitType					Not required.
PeakID	X	X	X	X	Report the peak identifier as used by the laboratory to uniquely identify this peak. This identifier must be consistent throughout an analytical sequence.
PercentDifference				X	Report the calculated Percent Difference for this peak to the nearest tenth of a percent.
PercentDifferenceLimitHigh				X	Report the upper limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitLow				X	Report the lower limit for the Percent Difference to the nearest tenth of a percent.
PercentDifferenceLimitType				X	Report "Method".
PercentRecovery					Not required.
PercentRecoveryLimitHigh					Not required.
PercentRecoveryLimitLow					Not required.
PercentRecoveryLimitType					Not required.
PercentRecoveryType					Not required.
PercentRSD		X			Report the calculated %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitHigh		X			Report the upper limit for the %RSD to the nearest tenth of a percent under the AnalysisGroup only.
PercentRSDLimitLow					Not required.
PercentRSDLimitType		X			Report "Method".
Resolution	X	X	X		For pesticides, report the percent resolution for midpoint INDA, INDB, or INDC initial calibration standards only. Report resolutions for all PEMs used in initial and calibration verification standards.
ResolutionLimitHigh					Not required.

TABLE 5. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: IPC	ICAL	CCV	FLO/GPC	Instructions
ResolutionLimitLow	X	X	X		Report the lower limit for the percent resolution.
ResolutionLimitType	X	X	X		Report "Method".
ResolutionType	X	X	X		Report "Percent_Resolution".
ResolutionUnits	X	X	X		Report "Percent".
Result					Not required.
ResultLimitHigh					Not required.
ResultLimitLow					Not required.
ResultLimitType					Not required.
ResultType					Not required.
ResultUncertainty					Not required.
ResultUnits					Not required.
RRF					Not required.
RRFLimitLow					Not required.
RRFLimitType					Not required.
TailingFactor					Not required.
TailingFactorLimitHigh					Not required.
TailingFactorLimitType					Not required.
Wavelength					Not required.
WavelengthUnits					Not required.
WeightingFactor					Not required.
PeakComparison					Not required.

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TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2a" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "2" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report the software version number.
Lab Contract	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	Report "Pest" or "Aroclor" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID						Report the Agency-assigned Lab Code.
Lab Name	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.
SamplePlusMethod	X	X	X	X	X	
ClientID	X	X				Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ClientMethodCode	X	X	X	X		Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID	X	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	Report "GCECD_External_Standard".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName						Not required.
ClientSampleID	X	X	X	X		Report the EPA Sample Number.
CollectedDate	X	X				Report the date and time the sample was collected.
CollectedEndDate						Not required.
Comment						Not required.
Composite						Not required.
CoolerID						Not required.
CustodyID	X	X				Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch						Not required.
Filtered						Not required.
LabContract	X	X	X	X		Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X				Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID						Not required.
LocationName						Not required.
MatrixID	X	X	X	X	X	Report "Water" or "Soil" as applicable.
MatrixMedium	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable.
MethodBatch						Not required.
MethodCategory						Not required.
MethodCode						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodLevel	X	X				Report "Low".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	Report "GC".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X				Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID						Not required.
PhaseAnalyzed						Not required.
Preservative	X	X				Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X		Report the Case Number.
ProjectName						Not required.
QCCategory		X	X	X		Report "Blank" for MB, LEB, IB, or CB; "Spike" for MS; "Spike_Duplicate" for MSD; or "Blank_Spike" for LCS.
QCLinkage		X	X	X		Report "LabReportingBatch" for MS/MSD; "PreparationBatch" for MB and LCS; "AnalysisBatch" for IB; or "CleanupBatch" for CB.
QCType	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "Instrument_Blank" for IB; "PT_Sample" for Performance Evaluation samples or Proficiency Testing samples; "Method_Blank" for MB; "Leachate_Extraction_Blank" for LEB; "Cleanup_Blank" for CB; "Matrix_Spike" for MS; "Matrix_Spike_Duplicate" for MSD; "Laboratory_Control_Sample" for LCS; or "Non_Client_Sample".
Quarantine	X					Report "Yes" or "No" based on sampling information.
SamplingBatch						Not required.
ShippingBatch						Not required.
SiteID						Not required.
SiteName						Not required.
StorageBatch						Not required.
Characteristic	X	X	X	X		
CharacteristicType	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH".

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
CharacteristicValue	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X		Report "C" for "Temperature".
Comment						Not required.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.
LabZipCode	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID						Not required.
AnalysisType	X	X	X	X		Report "Initial", "Dilution-01", "Reanalysis-01", or "Reinjection-01", then increment as necessary.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
Analyst	X	X	X	X		Report the Analyst's initials.
AnalyzedAmount	X	X	X	X		Report the volume of final extract added to the sample vial in microliters to at least two significant figures.
AnalyzedAmountUnits	X	X	X	X		Report "uL".
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID	X	X	X	X		Report the full EPA Sample Number with applicable suffixes per the requirements in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodCode				X		Not required.
ClientMethodID	X	X	X	X	X	Report "SOM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
Column	X	X	X	X		Report the GC Column used.
ColumnInternalDiameter	X	X	X	X		Report the GC Column Internal Diameter in millimeters.
ColumnInternalDiameterUnits	X	X	X	X		Report the Column Length in meters.
ColumnLength	X	X	X	X		Report "m".
ColumnLengthUnits						Not required.
Comment						Not required.
ConfirmationAnalysisID						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectorID						Not required.
DetectorType	X	X	X	X		Report "ECD".
DilutionFactor	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
InjectionVolume	X	X	X	X		Report the injection volume in microliters. Report volume to at least two significant figures.
InjectionVolumeUnits	X	X	X	X		Report "uL".
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	Report the Lab File ID.
LabFileID	X	X	X	X	X	Report the Lab File ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch						Not required.
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis	X	X	X	X		Report "Dry" for soil/sediment samples. Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids.
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.
AnalysisGroup						Not required.
Handling						Not required.
ReportedResult	X	X	X	X		
AnalysisGroupID						Not required.
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
AnalyteType	X	X	X	X		Report "Target" for all target analytes or "Spike" for all target analytes designated as spike analytes for MS/MSD and LCS analysis.
BiasErrorRatio						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Numbers as it appears in the SOW.
ClientAnalyteID	X	X	X	X		Report CAS Number.
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit						Not required.
ClientDetectionLimitUnits						Not required.
ClientQuantitationLimit	X	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Comment						Not required.
DetectionLimit	X	X	X	X		For target analytes, report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
DifferenceErrorRatio						Not required.
ExpectedResult						Not required.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits						Not required.
LabAnalysisID	X	X	X	X		Report the Lab File ID from the analysis this reported result was derived from.
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-OR.
LabResultStatus	X	X				Report "Preliminary" or "Final" as applicable.
PeakID						Not required.
PercentDifference	X	X	X	X		For Confirmation analyses, report the Percent Difference between the reported results and the confirmation results to the nearest whole percent (excluding IB).

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
PercentDifferenceLimitHigh	X	X	X	X		Report the upper limit for the Percent Difference to the nearest whole percent (excluding IB).
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType	X	X	X	X		Report "Method" (excluding IB).
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
QuantitationLimit	X	X	X	X		For target analytes, report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
RetentionTime						Not required.
RetentionTimeUnits						Not required.
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
RPDType						Not required.
PreparationPlusCleanup	X	X	X	X		
AliquotAmount	X	X	X	X		Report the sample amount in grams for soil/sediment or milliliters for aqueous/water to at least three significant figures.
AliquotAmountUnits	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water.
Analyst	X	X	X	X		Report the Analyst's initials.
CleanedUpDate	X	X	X	X		Report the date and time the sample was cleaned up.
CleanupBatch	X	X	X	X		Links all samples that were cleaned up together. Report the Lab File ID of the associated blank or other unique identifier.
CleanupType	X	X	X	X		Report "GPC", "Florisil", "Sulfur", or "Sulfuric_Acid" as applicable.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X		Report the month and year the SOW was issued.
Comment						Not required.
FinalAmount	X	X	X	X		Report the volume of material produced upon completion of this Prep or Cleanup in microliters.
FinalAmountUnits	X	X	X	X		Report "uL".
InitialAmount	X	X	X	X		Report the initial amount of extracted sample used for this cleanup method in microliters.
InitialAmountUnits	X	X	X	X		Report "uL".
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
LotNumber	X	X	X	X		Report the manufacturer's lot number for the Florisil cartridges used.
MethodCode						Not required.
MethodID	X	X	X	X		Report "SOM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
MethodSource	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X	X		Links all samples that were prepared together. Report the Lab File ID of the associated Method Blank.
PreparationPlusCleanupType	X	X	X	X		Report "Preparation" or "Cleanup" as applicable.
PreparationType	X	X	X	X		Report "Sonication", "Soxhlet", or "Pressurized_Fluid" for soil/sediment. Report "Sep_Funnel", "Liq_Liq", or "Liq_Membrane" for aqueous/water. Report "Waste_Dilution" for waste dilution.
PreparedDate	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.
Solvent						Not required.
Analyte	X	X	X	X		
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X		Report "Target" for all target analytes; "Spike" for all target analytes designated as spike analytes for MS/MSD or LCS analysis; or "Surrogate" for surrogate standards.
BiasErrorRatio						Not required.
CASRegistryNumber	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X		Report CAS Number.
ClientAnalyteName	X	X	X	X		Report the analytes as they appear in the SOW.
Comment						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectionLimit	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult	X	X	X	X		Report the theoretical final calculated concentration for MS/MSD and LCS. Report surrogates in nanograms.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
Inclusion						Not required.
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard.
PeakID	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentRecovery	X	X	X	X		Report the final calculated Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitHigh	X	X	X	X		Report the upper limit for the Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitLow	X	X	X	X		Report the lower limit for the Percent Recovery of the spikes and surrogates to the nearest whole percent.
PercentRecoveryLimitType	X	X	X	X		Report "Method".
PercentRecoveryType						Not required.
QuantitationLimit	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result	X	X	X	X		Report the final calculated concentration or amount to at least two significant figures. Leave blank if compound is not detected.
ResultLimitHigh						Not required.

TABLE 6. GAS CHROMATOGRAPHY DATA ELEMENT INSTRUCTIONS (Con't)

Node and Data Elements	Applicability: Sample	MS/MSD	LCS	MB/LEB/IB/CB	NCS	Instructions
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits	X	X	X	X		Report "ug/kg" for soil/sediment or "ug/L" for aqueous/water (or "mg/L" for TCLP).
StandardSource	X	X	X	X		Report the vendor/manufacturer for this standard.
Wavelength						Not required.
WavelengthUnits						Not required.
AnalyteGroup						Not required.

TABLE 7. ABBREVIATIONS

Abbreviation	Definition
%D	Percent Difference
%RSD	Percent Relative Standard Deviation
CAS	Chemical Abstracts Service
CB	Cleanup Blank
CCV	Continuing Calibration Verification
CRQL	Contract Required Quantitation Limit
DMC	Deuterated Monitoring Compound
DTD	Document Type Definition
EDD	Electronic Data Deliverable
FLO	Florisil Cartridge Check
GC	Gas Chromatography or Gas Chromatograph
GPC	Gel Permeation Chromatography Calibration Verification
IB	Instrument Blank
ICAL	Initial Calibration
ICV	Initial Calibration Verification
ID	Identifier
IPC	Instrument Performance Check
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MB	Method Blank
MS	Matrix Spike or Mass Spectrometer or Mass Spectrometry
MSD	Matrix Spike Duplicate
NCS	Non-Client (ZZZZZ) Sample
PAH	Polynuclear Aromatic Hydrocarbon
PEM	Performance Evaluation Mixture
QATS	Quality Assurance Technical Support
QC	Quality Control
RESC	Resolution Check Mixture
RPD	Relative Percent Difference
RRF	Relative Response Factor
SB	Storage Blank
SDG	Sample Delivery Group
SPLP	Synthetic Precipitation Leaching Procedure
SOW	Statement of Work
SVOA	Semivolatile Organic Analyte
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
VOA	Volatile Organic Analyte

APPENDIX A – FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

1.0 FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

The Method Detection Limit (MDL) study data deliverable consists of a Microsoft® Excel spreadsheet containing the following columns (Table A-1) in the order specified.

The "Required" field in Table A-1 identifies the columns that are always required to be populated.

The Contractor shall provide one spreadsheet for each combination of instrument ID, analytical method, and preparation method used to report results under this contract.

The Contractor shall deliver the spreadsheets to the recipients specified in Table 1 of Exhibit B – Reporting and Deliverables Requirements.

The format for the Microsoft® Excel file name shall be MDL_#.xls, where # can be any naming convention selected by the Contractor.

TABLE A-1. MDL STUDY DATA DELIVERABLE

Column	Required	Instruction
LabID	X	Report the agency-assigned Lab Code.
LabContract	X	Report the Lab Contract Number per the instructions for Header/LabContract.
MethodSource	X	Report the SOW per the instructions for SamplePlusMethod/ClientMethodID.
Method	X	Report the analytical method per the instructions for Header/LabDataPackageName.
PreparationMethod		Report the preparation method per the instructions for PreparationPlusCleanup/ClientMethodID.
ClientMethodCategory		Report the subset analyzed per the instructions for SamplePlusMethod/ClientMethodCategory if applicable.
ClientMethodModificationID		Report the MA number per the instructions for SamplePlusMethod/ClientMethodModificationID if applicable. Otherwise leave null.
Level		Report the sample level per the instructions for SamplePlusMethod/MethodLevel.
Matrix	X	Report the sample matrix per the instructions for SamplePlusMethod/MatrixID.
InstrumentID	X	Report the instrument ID per the instructions for Analysis/InstrumentID.
ColumnID	X	Report the GC column ID per the instructions for Analysis/Column if applicable.
ClientAnalyteID	X	Report the analyte per the instructions for ReportedResult/ClientAnalyteID.
Peak	X	Report the Peak ID per the instructions for Peak/PeakID.

Column	Required	Instruction
ResultUnits	X	Report the units for the replicate concentrations reported per the instructions for ReportedResult/ResultUnits.
Replicate##	X	The Laboratory shall include as many columns as there are replicates reported. Usually this would be seven, but more than seven replicates can be reported. The Laboratory shall report the results of the analysis of each replicate for each analyte. Each column shall be labeled "Replicate##", where the ## shall be replaced with the numeric designation of the replicate (e.g., Replicate01 for the first, Replicate02 for the second, Replicate03 for the third, etc.).
LabAnalysisID##	X	Following each Replicate## column, the Laboratory shall report a LabAnalysisID## column, reporting the LabAnalysisID of that replicate for that analyte per the instructions for Analysis/LabAnalysisID. The LabAnalysisID## columns shall be labeled in the same manner as the Replicate## columns.
AnalyzedDate##	X	Following each LabAnalysisID## column, the Laboratory shall report a AnalyzedDate## column, reporting the analysis date and time for that replicate for that analyte per the instructions for the Analysis/AnalyzedDate data element. The AnalyzedDate## columns shall be labeled in the same manner as the Replicate## columns. (MMDDYYYYThh:mm:ss)
StandardDeviation	X	Report the calculated standard deviation of the replicates for each analyte to at least three significant figures.
StudentsTValue	X	Report the appropriate Student’s T value for the degrees of freedom based on the number of replicates and 99% for the one-sided test.
DetectionLimit	X	Report the calculated Detection Limit for each analyte per the instructions for Analysis/Analyte/DetectionLimit.
DetectionLimitUnits	X	Report the appropriate units for the preparation method per the instructions for Analysis/Analyte/DetectionLimitUnits.
MDLAcceptable	X	Enter "Y" if the calculated MDL is less than the CRQL for the analyte and matrix. Otherwise enter "N".
ExpectedResult	X	Report the concentration for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResult.
ExpectedResultUnits	X	Report the concentration units for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResultUnits.

Column	Required	Instruction
ConcentrationAcceptable	X	Enter "Y" if the concentration of the analyte in the MDL standards was less than or equal to 10 times the calculated MDL for that analyte. Otherwise enter "N".
EffectiveDate	X	Report the date on which the Laboratory began to use the calculated MDL for reporting sample results for that analyte, instrument, and method formatted per the instructions for Header/DateFormat. This date cannot precede the analysis date of the MDL replicates.

Summary of Changes: SOM02.3 to SOM02.4

The following Summary of Changes highlights the major modifications implemented in SOW SOM02.4 compared to SOW SOM02.3.

This is a high-level summary and is not intended to be a complete or comprehensive listing of every modification. Interested parties are strongly encouraged to read the complete SOW SOM02.4 and familiarize themselves with all of the requirements.

Global

- References to "SOM02.3" have been updated to "SOM02.4".

Exhibit A

- **Section 5.4.4.6** – Requirements that the daily check of the infrared (IR) temperature detection device be documented and records maintained on file have been added.

Exhibit B

- **Section 2.2.1** – The instructions for data resubmission have been updated to indicate that corrected data submitted as "Additional Data" at the request of an EPA Regional data reviewer shall only include the affected pages and be accompanied by a revised Sample Delivery Group (SDG) Narrative documenting the reason(s) for the resubmittal.
- **Sections 2.4.7.2.4.3, 2.4.7.3.3, 2.4.8.2.4.3, 2.4.8.3.3, 2.4.9.2.4.3, and 2.4.9.3.3** – Language in these sections has been updated to specify that in all instances where manual integration or quantitation has been performed, the hardcopy printout(s) of the Extracted Ion Current Profiles (EICPs) of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s).
- **Sections 2.4.7.3.1, 2.4.7.3.2, 2.4.8.3.1, 2.4.8.3.2, 2.4.9.3.1, and 2.4.9.3.2** – Language in these sections has been updated to specify that the corresponding original system integration shall be included in the raw data, in addition to the EICPs displaying each manual integration.
- **Sections 2.4.7.3.2, 2.4.8.3.2, and 2.4.9.3.2** – Requirements have been specified for the submission of Form 7A-OR and associated raw data for the alternate source Initial Calibration Verification (ICV) standard for the Gas Chromatograph/Mass Spectrometer (GC/MS) methods.
- **Sections 2.4.10.2.4.2, 2.4.10.3.3, 2.4.11.2.4.2, and 2.4.11.3.3** – Language in these sections has been updated to specify that in all instances where manual integration or quantitation has been performed, the hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s).
- **Sections 2.4.10.3.1, 2.4.10.3.2, 2.4.11.3.1, and 2.4.11.3.2** – Language in these sections has been updated to specify that the corresponding original system integration shall be included in the raw data, in addition to the chromatograms displaying each manual integration.
- **Section 2.6.2.1.2, Table 3** – "Initial Calibration Verification" has been added to the list of child bookmarks associated with the Trace Volatile, Low/Medium Volatile, and Semivolatile Standards Data parent bookmark.

- **Section 2.7.1** – "I certify that this data package is..." has been updated to "I certify that these Preliminary Results are…" in the statement on the SDG Cover Page that is to be submitted with the Preliminary Results.
- **Section 3.3.7.1, Table 5** – Table 5 (Codes for Labeling Data) has been updated to clarify that laboratory QC samples not part of the SDG are to be reported as "ZZZZZ"; Language for Footnote 6 has been added to clarify that instrument QC samples must not be reported as "ZZZZZ".
- **Section 3.3.7.1, Table 5** – EPA Sample Number formats have been specified for the Volatile and Semivolatile alternate source ICV standards.
- **Section 3.3.22** – "WDIL" has been included as the code for reporting the Waste Dilution extraction type on the applicable Summary Forms.
- **Section 3.4.2.2.9** – Form 1A-OR and Form 1B-OR reporting requirements have been updated as follows and a Note added:

  Under column "Concentration", enter for each analyte, the value of the result if the concentration or mass is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions. If the concentration is less than the MDL, enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions. The concentration or mass result shall be reported to two significant figures.

  NOTE: For analytes in a sample that require more than one dilution, the compliant result from the least diluted analysis shall be considered as the best analytical result for the sample. For analytes in a sample that require dilution, reanalysis, or re-extraction, when none of the results from these analyses are compliant, the result from the initial analysis shall be considered as the best analytical result for the sample. For non-detected analytes that do not require any further dilution, reanalysis, or re-extraction, the CRQLs from the initial analysis shall be considered as the best analytical result.
- **Section 3.4.9.2.2** – The reporting instructions for the date(s) of the initial calibration of the single component pesticides analytes on Form 6B-OR and Form 6C-OR have been clarified as follows: Enter the dates of the first and the last initial calibration (ICAL) standard analyses in the entire ICAL sequence [excluding the Resolution Check Standard (RESC), Performance Evaluation Mixture standard (PEM), and instrument blanks].

  Note that the language in **Sections 3.4.14.2.1, 3.4.15.2.1, 3.4.16.2.1, and 3.4.18.2.1** has been updated to clarify that the dates specified in Section 3.4.9.2.2 above are also to be entered on Form 7B-OR, Form 7C-OR, Form 7D-OR, and Form 8B-OR.
- **Section 3.4.9.2.3** – The reporting instructions for the time(s) of the initial calibration of the single component pesticides analytes on Form 6B-OR and Form 6C-OR have been clarified as follows: Enter the times of the first and the last ICAL standard analyses in the entire ICAL sequence (excluding the RESC, PEM, and instrument blanks).
- **Section 3.4.13 and Associated Subsections** – Instructions have been provided for reporting the GC/MS alternate source ICV standard data on Form 7A-OR.

Exhibit B – Forms

- **Form 7A-OR** – "Initial Calibration Verification" has been added to the form title. The new title is "Initial Calibration Verification and Continuing Calibration Verification for GC/MS".

- **Form DC-2** – The Form 7A-OR title has been updated to "Initial Calibration Verification and Continuing Calibration Verification for GC/MS" for Categories 16, 32, and 48 under the Trace Volatiles, Low-Medium Volatiles, and Semivolatiles Standards Data sections, respectively.

Exhibit C

- **Section 3.0, Table 3** – Hexachlorobenzene and Pentachlorophenol have been designated as Toxicity Characteristic Leaching Procedure (TCLP) analytes.
- **Section 4.0, Table 4** – Endrin has been designated as a TCLP analyte.

Exhibit D – Introduction

- **Section 4.0** – Language has been added stating that stock solutions that are past the manufacturer's expiration date shall not be used to prepare analytical standards.

Exhibit D – General

- **Section 6.2.5** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Section 7.1.1** – The reagent water requirements have been updated to include the following: For the preparation of pH buffer solutions, it may be necessary to boil and cool the water prior to use.
- **Section 8.3** – The contract holding time requirements have been clarified as follows: The holding time for ZHE extraction of volatile soil samples or waste samples containing $\geq$ 0.5% solids is 10 days from Validated Time of Sample Receipt (VTSR). The holding time for TCLP or SPLP extraction of non-volatile soil samples or waste samples containing $\geq$ 0.5% solids is 10 days from VTSR. The holding time for TCLP or SPLP filtration of aqueous samples is 5 days from VTSR.

Exhibit D – Trace VOA

- **Sections 6.3 and 9.0 and Associated Subsections** – The requirements for analytical instrumentation and instructions for instrument calibration/standardization have been updated, wherever applicable, to include the alternate source ICV standard.
- **New Sections 7.2.2.2 and 9.4** – Instructions and requirements have been provided for the preparation and analysis of an ICV standard that is to consist of a solution from a different source or lot than that used for the ICAL standard.
- **Section 7.2.2.4** – The instructions for the preparation of the Deuterated Monitoring Compound (DMC) spiking solution have been updated to specify that the DMCs are to also be added to alternate source ICV standard.
- **New Section 9.5.6.4** – The corrective action for sample reanalysis is not required when the noncompliant analytes or associated Deuterated Monitoring Compounds (DMCs), in the opening or closing Continuing Calibration Verification (CCV) standards bracketing a dilution or a reanalysis, are not the same analytes or associated DMCs for which the dilution or reanalysis was intended.
- **Section 11.0 and Associated Subsections** – The Data Analysis and Calculations requirements have been updated, wherever applicable, to include the alternate source ICV standard.

- **Section 11.2.1.6** – Language has been updated to specify that in all instances where manual integration or quantitation has been performed, the hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s).
- **Section 11.4.4 and Associated Subsections** – The corrective actions to be taken when the DMCs and/or Internal Standards (ISs) do not meet the technical acceptance criteria in a sample have been clarified.
- **New Section 11.4.8** – The Sample Management Office (SMO) shall be contacted if the required corrective actions for sample reanalysis and/or dilution cannot be performed due to insufficient sample volume.
- **Section 12.0 and Associated Subsections** – The Quality Control requirements have been updated, wherever applicable, to include the alternate source ICV standard.
- **Section 17.0, Table 4** – The technical acceptance criteria (Minimum RRF, Maximum %RSD, and Maximum %D) for the alternate source ICV standard have been included in the table.

Exhibit D – Low/Medium VOA

- **Sections 6.3 and 9.0 and Associated Subsections** – The requirements for analytical instrumentation and instructions for instrument calibration/standardization have been updated, wherever applicable, to include the alternate source ICV standard.
- **New Sections 7.2.2.2 and 9.4** – Instructions and requirements have been provided for the preparation and analysis of an ICV standard that is to consist of a solution from a different source or lot than that used for the ICAL standard.
- **Section 7.2.2.4** – The instructions for the preparation of the Deuterated Monitoring Compound (DMC) spiking solution have been updated to specify that the DMCs are to also be added to alternate source ICV standard.
- **Section 8.3** – The contract required holding time requirements have been clarified as follows: Analysis of water and soil/sediment samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). Analysis of unpreserved, unfrozen soil/sediment samples must be completed within 24 hours of VTSR. The holding time for the analysis of TCLP or SPLP filtrates and leachates is 7 days from the completion of the TCLP or SPLP filtration and extraction procedures.
- **New Section 9.5.6.4** – The corrective action for sample reanalysis is not required when the noncompliant analytes or associated DMCs, in the opening or closing CCVs bracketing a dilution or a reanalysis, are not the same analytes or associated DMCs for which the dilution or reanalysis was intended.
- **Section 11.0 and Associated Subsections** – The Data Analysis and Calculations requirements have been updated, wherever applicable, to include the alternate source ICV standard.
- **Section 11.2.1.6** – Language has been updated to specify that in all instances where manual integration or quantitation has been performed, the hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s).

- **Section 11.4.4 and Associated Subsections** – The corrective actions to be taken when the DMCs and/or ISs do not meet the technical acceptance criteria in a sample have been clarified.
- **New Section 11.4.8** – SMO shall be contacted if the required corrective actions for sample reanalysis and/or dilution cannot be performed due to insufficient sample volume.
- **Section 12.0 and Associated Subsections** – The Quality Control requirements have been updated, wherever applicable, to include the alternate source ICV standard.
- **Section 17.0, Table 4** – The technical acceptance criteria (Minimum RRF, Maximum %RSD, and Maximum %D) for the alternate source ICV standard have been included in the table.

Exhibit D – SVOA

- **Section 6.1.12** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Sections 6.3 and 9.0 and Associated Subsections** – The requirements for analytical instrumentation and instructions for instrument calibration/standardization have been updated, wherever applicable, to include the alternate source ICV standard.
- **New Sections 7.2.2.2 and 9.4** – Instructions and requirements have been provided for the preparation and analysis of an ICV standard that is to consist of a solution from a different source or lot than that used for the ICAL standard.
- **Section 7.2.2.7.1** – The third sentence regarding the preparation of the internal standard spiking solution has been updated to: Just prior to full scan analysis by GC/MS, add sufficient amount of the internal standard spiking solution to an aliquot of the water, low-level, or medium-level soil sample extracts <u>for the initial analysis, dilution, and reanalysis, or to the re-extracts if applicable</u>, to result in a 20 ng/µL concentration of each internal standard.
- **Section 7.2.2.7.2** – The first sentence regarding the preparation of the internal standard spiking solution has been updated to: If the optional analysis of PAHs and PCP using the SIM analysis is to be performed, the Contractor shall add sufficient amount of the internal standard spiking solution to an aliquot of the water or low-level sample extracts <u>for the initial analysis, dilution, and reanalysis, or to the re-extracts if applicable</u>, just prior to SIM analysis to result in a 0.40 ng/µL concentration of each internal standard.
- **New Section 9.5.6.4** – The corrective action for sample reanalysis is not required when the noncompliant analytes or associated DMCs, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or associated DMCs for which the dilution, re-extraction, or reanalysis was intended.
- **Section 11.0 and Associated Subsections** – The Data Analysis and Calculations requirements have been updated, wherever applicable, to include the alternate source ICV standard.
- **Section 11.2.1.3** – Language has been updated to specify that in all instances where manual integration or quantitation has been performed, the hardcopy printout(s) of the EICPs of the quantitation ion displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the EICPs of the quantitation ion displaying the manual integration(s).
- **Section 11.4.4 and Associated Subsections** – The corrective actions to be taken when the DMCs and/or ISs do not meet the technical acceptance criteria in a sample have been clarified.

- **New Section 11.4.6** – SMO shall be contacted if the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume.
- **Section 12.0 and Associated Subsections** – The Quality Control requirements have been updated, wherever applicable, to include the alternate source ICV standard.
- **Section 17.0, Table 3** – Target analytes Hexachlorocyclopentadiene associated with 2,4-Dichlorophenol-d_3 (DMC-9) and 3,3'-Dichlorobenzidine associated with Benzo(a)pyrene-d_{12} (DMC-17) have been deleted from this table so that they are only associated with Nitrobenzene-d_5 (DMC-7) in this table.
- **Section 17.0, Table 5** – The technical acceptance criteria (Minimum RRF, Maximum %RSD, and Maximum %D) for the alternate source ICV standard have been included in the table.
- **Section 17.0, Table 10** – DMC 4, 6-Dinitro-2-methylphenol-d_2 associated with Internal Standard Phenanthrene-d_{10} has been deleted, and DMC 2,4-Dichlorophenol-d_3 has been added under Internal Standard Naphthlene-d_8 for consistency with Section 17.0, Tables 3 and 9.

Exhibit D – PEST

- **Section 6.1.12** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Section 8.3.1** – The contract required holding time requirements have been clarified as follows: Extraction of water samples by separatory funnel procedures must be completed within 5 days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction must be started within 5 days of VTSR. Extraction of the TCLP or SPLP filtrates and leachates shall begin within 7 days of completion of the filtration and leaching procedures. Extraction of soil/sediment samples shall be completed within 10 days of VTSR.
- **New Section 9.4.6.9** – The corrective action for sample reanalysis is not required when the noncompliant analytes or surrogates, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or surrogates for which the dilution, re-extraction, or reanalysis was intended.
- **Section 10.3.1.4.4** – The Percent Recovery (%R) limits of each analyte in the Gel Permeation Chromatography (GPC) calibration verification standard have been updated from "80-120%" to "80-110%" for consistency with the values in Section 17.0, Table 9.
- **Section 11.2.1.3** – The following Note has been added: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 4 – Pesticides Target Analyte List and Contract Required Quantitation Limits, and surrogates.

- **New Section 11.4.6** – SMO shall be contacted if the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume.

Exhibit D – ARO

- **Sections 6.1.12 and 6.2.12.1** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **New Section 9.4.6.9** – The corrective action for sample reanalysis is not required when the noncompliant analytes or surrogates, in the opening or closing CCVs bracketing a dilution, a re-extraction, or a reanalysis, are not the same analytes or surrogates for which the dilution, re-extraction, or reanalysis was intended.
- **Section 10.1.1.2.1** – The order of pH adjustment and addition of solvent has been changed.
- **Section 11.2.1.3** – The following Note 2 has been added: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC instrument operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the properly scaled raw chromatogram that clearly shows the manual integration. The GC instrument operator shall also mark each integrated area with the letter "m" on the quantitation report, and initial and date the changes. The hardcopy printout(s) of the chromatograms displaying the original integration(s) shall be included in the raw data, in addition to the hardcopy printout(s) of the chromatograms displaying the manual integration(s). This applies to all target analytes listed in Exhibit C – Organic Target Analyte List and Contract Required Quantitation Limits, Table 5 – Aroclors Target Analyte List and Contract Required Quantitation Limits, and surrogates.
- **New Section 11.4.6** – SMO shall be contacted if the required corrective actions for sample re-extraction, reanalysis, and/or dilution cannot be performed due to insufficient sample volume.

Exhibit F

- **Section 8.2.2** – The language pertaining to the content of the raw data files that are to be submitted during electronic data audits has been updated to include the GC/MS alternate source ICV standard data.

Exhibit H

- **Section 2.2.2** – The rounding requirements have been clarified as follows: The values reported by the Contractor are used for data assessment. No raw data values in the SEDD files shall be rounded. The Contractor shall not use rounded intermediate values in calculating the final result, and no rounding shall be performed until reaching the final result.
- **Section 3.1.5** – The ICV has been added to the list of instrument QC samples that must be reported as an InstrumentQC node under each Header node.
- **Section 3.1.7** – The requirements for the Analysis node associated with an initial analysis, dilution, or reanalysis have been clarified as follows:

  Each SamplePlusMethod must contain at least one Analysis node for Gas Chromatograph/Mass Spectrometer (GC/MS) methods or must contain at least two Analysis nodes for GC methods with confirmation (one for each column). Separate Analysis nodes are required for each

dilution, re-extraction, or reanalysis. Any reanalysis must be preceded by an initial analysis. Any analysis reported as a dilution must also have a less-diluted analysis reported as initial. The initial analysis does not have to precede the diluted analysis.

Each Instrument QC node (other than Initial Calibration) must contain one Analysis node for GC/MS methods or must contain at least two Analysis nodes for GC methods with confirmation (one for each column).

- **Section 7.0, Tables 1 and 2** – The "Applicability" column has been updated to include the GC/MS alternate source ICV standard.
- **Section 7.0, Tables 1, 2, and 3** – The Instructions for the ClientMethodCode element under the SamplePlusMethod node have been updated from "Not required" to "Report "PAH", "TCLP" or "SPLP" when applicable".
- **Section 7.0, Tables 4, 5, and 6** – The Instructions for the ClientMethodCode element under the SamplePlusMethod node have been updated from "Not required" to "Report "TCLP" or "SPLP" when applicable".
- **Section 7.0, Tables 1, 2, and 3** – The Applicability of the OriginalClientSampleID element under the SamplePlusMethod node has been revised to include the "Sample", and the Instructions have been updated to: Report the EPA Sample Number of the original sample this sample was derived from. Report the EPA sample number used for the low level sample analysis for the volatiles and semivolatiles medium level samples, if applicable. Leave blank if only the medium level analysis is performed for the sample.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – "Report "None" if sample was not preserved" has been added to the Instructions for the Preservative element under the SamplePlusMethod node.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – "Tissue samples do not require "Percent_Solids" or "pH"" has been added to the Instructions for the CharacteristicType element under the SamplePlusMethod/Characteristic node.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – The Instructions for the CharacteristicValue element under the SamplePlusMethod/Characteristic node have been updated to: For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – "Report "Wet" for tissue samples or for any other matrices for which the results are not corrected for percent solids" has been added to the Instructions for the ResultBasis element under the SamplePlusMethod/Analysis node.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – "Report "Waste_Dilution" for waste dilution" has been added to the Instructions for the PreparationType element under the SamplePlusMethod/ Analysis/PreparationPlusCleanup node.
- **Section 7.0, Tables 1, 2, 3, 4, 5, and 6** – The Instructions for the DetectionLimit element under the SamplePlusMethod/Analysis/Analyte node have been updated from "Report the MDL" to "Report the MDL to at least two significant figures".

- **Section 7.0, Tables 1 and 4** – "Unadjusted for sample weight/volume, percent solids, or dilution factor" has been added to the Instructions for the IntermediateResult element under the SamplePlusMethod/Analysis/Analyte node.
- **Section 7.0, Tables 1 and 2** – The Instructions for the ClientMethodCode element under the InstrumentQC node have been updated from "Not required" to "Report "PAH", "TCLP", or "SPLP" when applicable".
- **Section 7.0, Tables 4 and 5** – The Instructions for the ClientMethodCode element under the InstrumentQC node have been updated from "Not required" to "Report "TCLP" or "SPLP" when applicable".
- **Section 7.0, Tables 1, 2, 4, and 5** – The Instructions for the ClientMethodModificationID element under the InstrumentQC node have been updated from "Not required" to "Report the Modified Analysis Number, if applicable".
- **Section 7.0, Tables 1 and 2** – "Initial Calibration Verification for ICV" has been added to the Instructions for the QCType element under the InstrumentQC node.
- **Section 7.0, Tables 1 and 2** – The Instructions for the ExpectedResult element under the InstrumentQC/Analysis/Analyte node have been updated to "Report the final amount for all applicable target analytes, DMCs, and internal standards".
- **Section 7.0, Tables 4 and 5** – The Instructions for the ExpectedResult element under the InstrumentQC/Analysis/Analyte node have been updated to "Report the final amount for all applicable target analytes and surrogates".
- **Section 7.0, Tables 1 and 4** – "Unadjusted for sample weight/volume, percent solids, or dilution factor" has been added to the Instructions for the IntermediateResult element under the InstrumentQC/Analysis/Analyte node.
- **Section 7.0, Table 4** – "Leave blank if compound not detected" has been deleted from the Instructions for the IntermediateResult element under the InstrumentQC/Analysis/Analyte/Peak node.
- **Appendix A, Table A-1** – "ReportedResult/DetectionLimit" has been replaced with "Analysis/Analyte/DetectionLimit" in the Instructions for the "DetectionLimit" column in the MDL study data deliverable table.
- **Appendix A, Table A-1** – "ReportedResult/DetectionLimitUnits" has been replaced with "Analysis/Analyte/DetectionLimitUnits" in the Instructions for the "DetectionLimitUnits" column in the MDL study data deliverable table.

Appendix D

TO-15 SIM Analytical Reporting Limits

Eurofins Air Toxics - TO-15 SIM Reporting Limits

	1	2	3	4	5	6	7
1	**Method**	**CAS #**	**Analyte**	**RL ppbv**	**MDL ppbv**	**RL ug/m3**	**MDL ug/m3**
2	TO-15 SIM	71-55-6	1,1,1-Trichloroethane	0.02	0.001	0.11	0.005
3	TO-15 SIM	79-34-5	1,1,2,2-Tetrachloroethane	0.02	0.001	0.14	0.007
4	TO-15 SIM	79-00-5	1,1,2-Trichloroethane	0.02	0.002	0.11	0.011
5	TO-15 SIM	75-34-3	1,1-Dichloroethane	0.02	0.002	0.081	0.008
6	TO-15 SIM	75-35-4	1,1-Dichloroethene	0.01	0.003	0.04	0.012
7	TO-15 SIM	106-93-4	1,2-Dibromoethane (EDB)	0.02	0.001	0.15	0.008
8	TO-15 SIM	107-06-2	1,2-Dichloroethane	0.02	0.001	0.081	0.004
9	TO-15 SIM	106-46-7	1,4-Dichlorobenzene	0.02	0.002	0.12	0.012
10	TO-15 SIM	71-43-2	Benzene	0.05	0.005	0.16	0.016
11	TO-15 SIM	56-23-5	Carbon Tetrachloride	0.02	0.001	0.12	0.006
12	TO-15 SIM	75-00-3	Chloroethane	0.05	0.007	0.13	0.018
13	TO-15 SIM	67-66-3	Chloroform	0.02	0.002	0.01	0.01
14	TO-15 SIM	74-87-3	Chloromethane	0.05	0.012	0.1	0.025
15	TO-15 SIM	156-59-2	cis-1,2-Dichloroethene	0.02	0.002	0.079	0.008
16	TO-15 SIM	100-41-4	Ethyl Benzene	0.02	0.001	0.087	0.004
17	TO-15 SIM	76-14-2	Freon 114	0.02	0.001	0.14	0.007
18	TO-15 SIM	75-71-8	Freon 12	0.02	0.002	0.099	0.01
19	TO-15 SIM	108-38-3	m,p-Xylene	0.04	0.001	0.17	0.004
20	TO-15 SIM	1634-04-4	Methyl tert-butyl ether	0.1	0.002	0.36	0.007
21	TO-15 SIM	91-20-3	Naphthalene	0.05	0.014	0.26	0.073
22	TO-15 SIM	95-47-6	o-Xylene	0.02	0.001	0.087	0.004
23	TO-15 SIM	127-18-4	Tetrachloroethene	0.02	0.001	0.14	0.007
24	TO-15 SIM	108-88-3	Toluene	0.02	0.001	0.075	0.004
25	TO-15 SIM	156-60-5	trans-1,2-Dichloroethene	0.1	0.003	0.4	0.012
26	TO-15 SIM	79-01-6	Trichloroethene	0.02	0.002	0.075	0.011
27	TO-15 SIM	75-01-4	Vinyl Chloride	0.01	0.002	0.026	0.005
28							
29	MDL values vary by instrument. Values provided represent an approximate range.						
30	RL and MDL values do not account for sample dilution due to canister pressurization.						