STANDARD OPERATING PROCEDURE

FOR

VOLATILE ORGANICS BY METHOD 8260D

PHILIS SOP L-A-101 Rev. 3

Revision Date: 05-31-2024

EPA Contract No. 68HERH21D0002

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Revision History

Revision	Name	Date	Description of Change
A	James Travis	04/01/2021	Transitional Issue
0	James Travis	03/21/2022	Program Issue
1	James Travis	06/09/2022	Revision
2	James Travis	10/14/2022	Revision
3	James Travis	12/07/2023	Revision

SOP REVISION FORM

SOP Name:	**Volatile Organics by Method 8260D**		
Purpose: *(Review or Revise)*	*SOP #:*	*Rev. #:* *(Being Reviewed or Revised)*	*Origination / Release Date:*
Revision	*SOP No. L-A-101*	2	10/26/2022
Requested by:	James Travis	Date:	12/07/2023

New SOP Revision Date:	**05/31/2024**	**New SOP Revision #:** *(If Applicable)*	**3**

For *Revision* : Summary of Revisions (specify sections)

6.1	Corrected formatting Issues
7.1	Corrected formatting Issues
7.4	Corrected formatting Issues
7.1.3	Expiration passage removed from this section
7.1.13	Expiration passage removed from this section
7.5.2	Specific reagent amounts modified to reflect current procedure
7.5.5	Archon IS/SS is no longer used with this method, so passage was removed
7.5.6, 7.5.7	Passages removed due to redundancy with section 7.5.3 and 7.5.4
10.1.2.1	Masshunter added
11.3.10, 11.3.13	Specific reagent amounts modified to reflect current procedure
11.3.11	Autosampler name removed
17.0	Updated Figure 2 to current version of form

For *Review*: Comments

Standard Operating Procedure
Volatile Organics by Method 8260D
L-A-101 Rev. 3

TABLE OF CONTENTS

1.0 Scope and Application, Including Components to be Analyzed 1
2.0 Summary of Method 1
3.0 Definitions 3
4.0 Interferences 6
5.0 Health and Safety Warnings 7
6.0 Equipment and Supplies 7
6.1 Sampling Equipment for Aqueous Samples 7
6.2 Sampling Equipment for Soil/Solid Samples 8
6.3 Glassware 8
6.4 Syringes 8
6.5 Instrumentation 8
6.6 Equipment 9
6.7 Supplies 9
7.0 Reagents and Standards 9
8.0 Sample Collection, Preservation, Shipment and Storage 15
9.0 Quality Control and Acceptance Criteria 16
10.0 Calibration and Standardization 18
11.0 Procedure 20
12.0 Data Analysis and Calculations 24
13.0 Method Performance 28
14.0 Pollution Prevention 29
15.0 Waste Management 29
16.0 References 29
17.0 Tables, Diagrams, Flowcharts and Validation Data 31

TABLES AND FIGURES

Table 1. Examples of Analytes, MDLs, and RLs for Water using EPA Method 8260D 31
Table 2. Examples of Analytes, MDLs, and RLs for Soils using EPA Method 8260D 32
Table 3. Example Preparation of PDS, IS/SS, SCV-PDS in Methanol for Analyses of Aqueous Samples and Methanol Extractions 33
Table 4. Example Preparation of PDS, IS/SS, SCV-PDS methanol standards for Analyses of Soil/Solids 34
Table 5. Example Preparation of Aqueous Working Standards in Edison 34
Table 6. Example Preparation of Working Standards for Analyses of Low Level Soil/Solids 35
Table 7. Example Preparation of Aqueous Working Standards for Analyses of High Level Soils 35
Table 8. Recommended VOC Sample Preservation Techniques and Holding Times taken from SW 846 Method 5035A and Chapter 4 Table 4-1 36
Table 9. BFB Relative Abundance Suggested Criteria 38
Table 10. Example Relative Response Factor Criteria for Initial and Continuing Calibration Verification 38
Table 11. Example EPA Method 8260D Analysis Sequence with QC and Instrument Calibration Frequency and Acceptance Criteria 39
Table 12. Example Purge and Trap-GC/MS Settings for EPA Method 8260D 40
Table 13. Example Quantitation Ions and Qualifiers 42
Table 14. 8260D Method Acceptance Criteria 43
Figure 1. 50 µg/L 8260D Total Ion Chromatogram 44
Figure 2. Example GC/MS Data Review Form 45

Standard Operating Procedure
Volatile Organics by Method 8260D
L-A-101 Rev. 3

1.0 Scope and Application, Including Components to be Analyzed

1.1 This method can be used to determine the presence and concentration of the volatile analytes listed in Section 17 Tables 1 & 2 in aqueous samples: groundwater, surface water, wastewater, soil, solid, and waste samples.

1.2 This SOP is applied for purgeable organic analytes from aqueous or solid matrices except where a specific Quality Assurance Project Plan (QAPP) overrides this method's quality control and acceptance criteria.

1.3 This standard operating procedure (SOP) documents CSS's application of EPA Method 8260D, Revision 4, dated June 2018, EPA Method 5030C, Revision 3, May 2003-Determination of Volatile Organic Compounds in aqueous samples by Purge-and-Trap Gas Chromatography/Mass Spectrometry and EPA Method 5035A, Revision 1, July, 2002-Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, and EPA Method 3585, Revision 0, dated December 1996 that are used in the PHILIS Mobile Labs. This SOP may also be used with EPA Methods 5031, 5032, and 5041 which are not currently used in the PHILIS Mobile Labs.

1.4 This SOP is executed in accordance with the U.S. Environmental Protection Agency and National Environmental Laboratory Accreditation Program (NELAP).

1.5 Example detection limits for analytes determined by EPA Method 8260D are listed in Section 17 Tables 1 & 2 for waters and soils.

2.0 Summary of Method

2.1 Aqueous Samples (Based on SW846 Method 5030C): Helium is bubbled through 10 mL of water in a purge and trap sparge vessel. The purgeables are efficiently transferred from the liquid phase to the gaseous phase. The vapor is swept on a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

2.2 Soil Samples (Based on SW846 Method 5035A): Samples are collected and prepared in accordance with methods based on the potential level of volatile organic contaminants that are estimated to be present. Methods for low level VOCs are considered for samples that will generally fall in the 0.5 to 200 µg/kg range. Methods for High VOC concentrations (methanol extractions) are designed for samples estimated to contain VOC levels greater than 200 µg/kg.

2.2.1 Volatile Organic Compounds at low levels in soil samples are determined by collecting a 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed 40 mL VOA vial with a septum sealed screw-cap that already contains a stirring bar and sodium bisulfate preservation solution. The vial is sealed and transported to the PHILIS mobile labs. The entire vial is then placed, unopened, into the soil/water autosampler. Prior to analysis, organic-free reagent water, surrogates, and internal standards are automatically added to the sample by the OI 4100 soil/water autosampler without opening the vial. The vial is then heated to 40 °C, stirred and purged onto an adsorbent trap using helium carrier gas. When purging is then complete, the trap is then backflushed with helium and thermally desorbed onto the GC column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

2.2.2 An alternative to field preservation is the use of Encore Samplers (or equivalent) as collection and storage devices. Samples collected in this device must be preserved by the laboratory or analyzed within 48 hours of collection. The soil sample is removed with the use of an extrusion tool and analyzed or preserved. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer. Refer to Section 17 Table 8 for holding time and preservation requirements.

2.2.3 High level soils are extracted with methanol and analyzed as an aqueous sample. Typically, this would be approximately 5 grams of soil extracted with 5 mL of Methanol. A maximum of 100 uL (or 1mL spiked into 50mL – and 10mL of the 50mL purged) of methanol extract added to 5 mL reagent water should be purged in the system, as described above.

2.3 Oily Samples (SW 846 Method 3585): Samples that contain oily material are assessed to determine water-miscibility. Samples that are soluble in methanol may be weighed and diluted with methanol for analysis as in 5.1. Samples that are not soluble in water miscible solvents are prepared by dilution with hexadecane, or other suitable solvent, and directly injected for analysis. The gas chromatograph is temperature programmed to separate the components which are then detected with a mass spectrometer.

3.0 Definitions

3.1 Batch‡: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A Preparation Batch is composed of between 1 and 20 environmental samples of the same matrix, meeting the above criteria and with a maximum time between the start of processing of the first and the last sample in the batch to be 24hours. An Analytical Batch is composed of prepared environmental samples (extracts, digestates, or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples

An 8260 volatiles analytical batch will consist of no more than twenty (20) environmental samples in addition to the SOP Quality Control requirements.

All batches require one MB, LCS, and MS/MSD pair or MS and Sample Duplicate where possible.

3.2 BFB: 4-bromofluorobenzene or a solution that contains the analyte, 4-bromofluorobenzene, which is used to evaluate the tuning and the performance of the mass spectrometer. The BFB tune is required to be analyzed at the beginning of each 12-hour period during which a calibration is analyzed. A tune is not required for a sequence where a calibration is not performed but may be analyzed at the analysts discretion.

3.3 Chain of Custody (COC) ‡: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; preservation; and requested analyses.

Each time the samples are transferred, the document should be signed by the person releasing the samples and by the person receiving the samples. A date and time must also be recorded.

3.4 Continuing Calibration Verification (CCV): A standard analyzed at the beginning of each analytical sequence that contains all method analytes at a concentration near the mid-range of the calibration curve. Each analyte must have a recovery within a percentage range specified in the method to validate that analyte in the calibration curve. A CCV is not required if a calibration curve is analyzed at the start of an analysis sequence. Some methods require additional CCV's. The CCV frequency will be stated in the method SOP.

3.5 Holding Time: The maximum amount of time permitted between sampling and sample preparation and/or sample preparation and sample analysis. Also the period of time a sample may be stored prior to analysis when there is no preparation step. See the specific method or SW846 8000B Table 4.1.

3.6 Initial Demonstration of Proficiency (IDP): Also known as a Demonstration of Capability (DOC). A procedure involving the analysis of a calibration and QC samples to demonstrate precision and accuracy after method development, significant changes in instrumentation or after training a new analyst. Procedure outlined in Section 12.1.

3.7 Instrument Calibration Standards (ICS): A solution prepared from the primary dilution standard solution or stock standard solutions, internal standards and surrogate analytes. The ICS solutions are used to calibrate the instrument response with respect to analyte concentration.

3.8 Internal Standards (IS)[∓]: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

3.9 Laboratory Control Sample (LCS)[∓]: (however named, such as laboratory fortified blank, blank spike (BS), or QC check sample). A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

The standard source can be the same as the calibration or a second source. The LCS is analyzed exactly like a sample to determine whether the method is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.10 Laboratory Duplicate (LD): Two sample aliquots taken in the laboratory and analyzed separately with identical procedures. Analyses of the aliquots indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.11 Lower Limit of Quantitation (LLOQ): The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve. The laboratory shall establish the LLOQ at concentrations where both quantitative and qualitative criteria can consistently be met. Procedures for LLOQ verification are outlined in section 12.3.

3.12 Matrix Spike (spiked sample or fortified sample)[∓]: A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of the sample for which an independent test results of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

3.13 Matrix Spike Duplicate (spiked sample or fortified sample duplicate)‡: A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

3.14 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples of the same batch. Method Blank analytical results are evaluated to determine the presence of contamination in the analytical method process. A blank analyte result must be < ½ the LLOQ for the blank to be acceptable or results above the LLOQ must be greater than 10 times the blank concentration not to be flagged.

3.15 Method Detection Limit (MDL): The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. It is determined by analyzing seven or more replicates of a spiked analyte free matrix and the resulting statistical calculation, in accordance with 40 CFR 136, Appendix B, Revision 2.

3.16 Primary Dilution Standard (PDS): A solution of one or several analytes prepared in the laboratory from SSS and diluted as needed to prepare calibration solutions and other needed analyte solutions.

3.17 Quality Control Sample (QCS)‡: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.

3.18 Reporting Limit (RL): The reporting limit, also known as the LOQ is the minimum concentration that can be reported as a quantitated value for a target analyte in a sample. This value can be no lower than the concentration of the lowest calibration standard.

3.19 Required Detection Limit (RDL): Detection limits established by a client or regulatory authority for analytes of concern. The laboratory MDL values must be equal or lower than the RDL. This is also known as the CRQL, the contract-required quantitation limit.

3.20 Second Source Calibration Verification (SCV): A solution prepared from a source that is different from the calibration standards. The SCV is immediately following the ICS, and is used to verify calibration standard accuracy.

3.21 Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased as certified from a reputable commercial source.

3.22 Surrogate Standard (SS): Organic compounds which are similar to the target analytes in chemical composition and mimic the behavior of the target analytes throughout the analytical process. Surrogate compounds are not normally found in environmental samples. Each calibration standard, sample, MB, LCS, MS, and MSD is spiked with surrogate standards. Surrogates are used to evaluate analytical efficiency by measuring recovery. See analytical method SOP for a list of specific surrogate compounds that are appropriate for sample-specific analysis.

3.23 Working Standards (WS): Instrument calibration/calibration verification standards and quality control standards used in an analytical sequence such as BFB, ICS, CCV, SCV, MS, and MSD.

3.24 Trip Blank: An aliquot of reagent water placed in a VOA vial that travels with the cooler to determine if contaminants or interferences were introduced into the samples during sampling or transportation of the containers and samples

‡ EL-V1M2-ISO-2016, 2016 NELAP Standard definition.

4.0 Interferences

4.1 Samples for volatile organics analyses (VOA) are susceptible to laboratory chemical contaminants (e.g.: methylene chloride, acetone). Samples may become contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage.

4.2 Carryover contamination may occur when a sample containing low levels of VOCs is analyzed immediately following a sample containing high levels of VOCs. If this situation occurs during a non-monitored analysis, the sample containing the low concentration VOCs may require reanalysis. If the situation occurs during monitored analysis, a blank should be run to ensure that the system is free of contamination, and in addition, the sample should be re-analyzed at a higher dilution factor.

4.3 Other contamination or interferences could be present in laboratory glassware, chemicals, and reagents used.

4.4 Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution option in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect

low concentration samples in vials without chemical preservation. Samples of different preservation should be batched separately in the lab with QC reflecting the same preservation. Preservation should be documented accordingly on COC's and in LIMS.

4.5 Water samples may also effervesce upon addition of HCL preservative. Non-preserved samples, should be stored and batched separately from other samples and properly documented.

4.6 Mobile laboratories with volatile analysis areas must be kept at positive pressure to keep from drawing contaminants into the area and instruments.

5.0 Health and Safety Warnings

5.1 This method does not address all safety issues associated with its use. Laboratory personnel are responsible for maintaining a safe work environment and a current awareness of the Chemical Hygiene Plan regarding the safe handling of the chemicals listed in this method.

WARNING: The following VOCs have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride.

5.2 The toxicity and/or carcinogenicity of the other reagents and analytes used in this method have been defined; however, each chemical and sample should be treated as a potential health hazard. Exposure should be reduced to the lowest possible level. Procedures involving primary standards and sample preparation should be performed in a fume hood.

5.3 Safety Data Sheets (SDS) for each analyte and reagent used in the mobile laboratory are available to all employees. SDS should be consulted prior to handling chemicals.

5.4 Laboratory personnel are required to be familiar with the general laboratory safety including the location and proper use of safety/emergency equipment.

5.5 Acid preservatives and preserved samples are corrosive and should be handled and disposed of accordingly.

6.0 Equipment and Supplies

6.1 Sampling Equipment for Aqueous Samples

6.1.1 40 mL pre-cleaned VOA vials fitted with Teflon-lined screw caps.

6.1.2 40 mL VOA vials preserved with HCl and fitted with Teflon-lined screw caps.

6.1.3 40 mL VOA vials preserved with 100 ul of 0.25 mg/ul ascorbic acid solution and fitted with Teflon-lined screw caps. 1:1 HCL to be added in the field.

6.1.4 40 mL VOA vials preserved with 50ul of 0.07 mg/ul Na2s3O3 solution and fitted with Teflon-lined screw caps.

6.2 Sampling Equipment for Soil/Solid Samples

6.2.1 Pre-weighed 40 mL pre-cleaned VOA vials containing 5 mL of 20% sodium bisulfate solution and magnetic stir bar fitted with Teflon-lined screw caps.

6.2.2 Pre-weighed 40 mL pre-cleaned VOA vials containing 5 mL of methanol and a magnetic stir bar fitted with Teflon-lined screw caps.

6.2.3 Pre-weighed 40 mL pre-cleaned VOA vials containing 5 mL of Tri-sodium phosphate solution and magnetic stir bar fitted with Teflon-lined screw caps for fuel oxygenates.

6.2.4 Pre-weighed 40 mL pre-cleaned VOA vials containing 5 mL of reagent water and a magnetic stir bar, fitted with Teflon-lined screw caps.

6.2.5 Encore® sampler.

6.2.6 4-oz or 8-oz soil jars for moisture determination.

6.3 Glassware

6.3.1 Volumetric flask- class A, various sizes.

6.3.2 Disposable Pasteur pipettes.

6.3.3 Mininert vials (2 mL or 5 mL) and Mininert valves or 2mL screw cap vials.

6.3.4 1-L or 2-L Erlenmeyer Flask(s).

6.3.5 OI Analytical 50mL or 25-mL sparge vessel.

6.3.6 OI 5mL or 10mL loop.

6.3.7 5 mL measuring pipette (for analyses of soils/solids).

6.4 Syringes

Gas-tight micro syringes – various sizes.

6.5 Instrumentation

6.5.1 Agilent 6890N Gas Chromatograph or equivalent.

6.5.2 Agilent 5973 Mass Spectrometer- electron impact only (70 eV) or equivalent.

6.5.3 OI Analytical Eclipse 4760 Purge and Trap Concentrator or equivalent.

6.5.4 OI Analytical 4100 sample processor or equivalent.

6.5.5 Agilent MSD ChemStation G1701 DA software (or higher revision) or equivalent.

6.5.6 Restek, RTX-VMS 20 m (L) x 0.18 mm (id) x 1.0 µm (d_f) gas chromatographic column or equivalent.

6.5.7 OI Analytical Trap #10 or equivalent.

6.5.8 NIST 2002 Mass Spectral library (or higher revision) or equivalent.

6.6 Equipment

6.6.1 Heated Stirrer.

6.6.2 Nitrogen purge line for reagent water.

6.6.3 Drying oven (for analyses of dry weight for soils /solids).

6.6.4 Moisture analyzer (for analyses of soils/solids).

6.7 Supplies

6.7.1 Magnetic stir bars.

7.0 Reagents and Standards

Records are retained for all standards and reagents including the manufacturer/vendor, the Manufacture's Certificate of Analysis or purity, the date of receipt, recommended storage conditions, and an expiration date. Standard and reagent preparations are documented in the LIMS system, which will track dilutions and trace them to purchased stocks by lot numbers or neat compounds, reference to the method of preparation, date of preparation, expiration date of prepared solution and preparer's initials.

7.1 Reagents

Original containers of reagents must be labeled with an expiration date. All containers of prepared reagents must bear a name, preparation date, and must be recorded in the LIMS system or in a preparation log

7.1.1 Organic Free Reagent Water – water that does not contain analytes of interest or interferences that would prevent detection of analytes of interest at the reporting limit.

Deionized water with resistivity of 18.2 MΩ-cm or greater, treated with heated nitrogen purge for at least 1 hour to eliminate organic contaminants

7.1.2 HCl 37% ACS Grade. For preparation of 1:1 HCl for sample preservation in the field, and batch QC preparation for samples that are preserved in the field.

7.1.3 Preparation of 1:1 HCl. Add 15 mL of reagent water to a 40mL vial. Carefully add 15mL 37%HCL to the water. Purge with Nitrogen for one hour. Two or three drops from a pastuer pipette per vial for field samples not undergoing dechlorination. Samples dechlorinated in the field with ascorbic acid must be acidified after adding to the vial with ascorbic acid. Retain vials with acid or a portion of the field added HCl for batch QC.

7.1.4 L Ascorbic Acid > 99%- For dechlorination of aqueous samples in the field where field acid preservation will be done in the field. 3mg solid ascorbic acid added to 40ml vials before shipment or use aqueous solution.

7.1.5 Preparation of 0.25 mg/uL aqueous solution of ascorbic acid: Add 62.5g ascorbic acid to a 250mL beaker. Add 100mL reagent water and stir to dissolve powder. Decant the liquid a 250mL volumetric and add water in smaller portions and stir until powder is completely dissolved. Decant portions into volumetric flask and bring up to mark with reagent water. Invert and decant into a 250mL amber wide mouth jar. Purge with Nitrogen for one hour, create LIMD ID and label. 100uL into a 40ml vial. 80uL per 50Ml volumetric flask for QC preparation.

7.1.6 Sodium Thiosulfate (Na2S2O3) ACS Grade: Added to vials for aqueous samples as solid (3mg) or in solution form for dichlorination of samples where dichlorination and cooling are the only preservation options chosen. Prepare batch QC using the same lot used to preserve the samples.

7.1.7 Preparation of 0.07 mg/uL NaS2O3 solution. Weigh 17.5 g of NaS2O3 into a clean beaker, add 100mL reagent water and stir to dissolve powder. Decant the liquid a 250 mL volumetric and add water in smaller portions and stir until powder is completely dissolved. Decant portions into volumetric flask and bring up to mark with reagent water. Invert and decant into a 250 mL amber wide mouth jar. Purge with Nitrogen for one hour, create LIMD ID and label. 50 uL of solution per 40mL vial. Retain at least 4 vials per 20 samples for QC preparation.

7.1.8 Methanol- Purge and Trap grade only. 5ml of methanol are added to 40ml vials where approximately 5 g of solid/soil samples are added in the field or collected in Encore sampler and added in the lab. In the case of field preservation in methanol, vials with solvent are tared in the lab where tare weights are recorded in a logbook and on the vial label in indelible ink. A balance check of calibration weights is done and recorded in the same log within a 12 hour time frame.

7.1.9 Hexadecane – ACS Reagent Grade

7.1.10 Helium carrier gas: 99.999% (UHP) grade or better.

7.1.11 Nitrogen- purge gas, 99.999% (UHP) grade or better.

7.1.12 Sodium bisulfate monohydrate (NaHSO4), 97% (or better) for acid preservation of soil/solid samples in the field or added to the contents of an Encore sampler in the lab.. One gram of solid NaHSO4 + 5ml reagent water per approximately 5g of sample in a 40ml vial with a magnetic stir bar or use 5ml of a 20% aqueous solution with a stir bar to the same.

7.1.13 Preparation of an aqueous 20% NaHSO4 solution: Carefully add 57.6 g of solid NaHSO4 crystals to 100ml reagent water in a clean 250 mL beaker with a plastic spoon or spatula.

Note: NaHSO4 crystals are acidic and highly corrosive. Stir until most of the crystals have dissolved and decant the liquid into a 250 mL volumetric flask. Add smaller amounts of water, stir and decant smaller portions of water until all the crystals have dissolved. Bring the solution to the 250 mL mark with reagent water, Invert flask and decant into s 250 mL wide mouth amber jar and purge with nitrogen for an hour. Check and periodically check the solution for interferences. Use 5 mL with a stir bar per 5 g of sample or blank sand.

7.1.14 Ottawa Sand- Reagent Grade (for analyses of soils/solids). Other sands may be used provided they do not contain analytes of interest or interferences that would prevent reporting analytes at their reporting limits. Purchased sand may require cleaning before use as reagent (blank) sand.

7.1.14.1 Preparation of Reagent Sand option 1 (Edison/preferred)- Check lab in building 209 for muffle furnace availability. Add raw Ottawa sand into a one liter beaker or multiple one liter beakers. Bake for four hours or overnight in the muffle furnace at 400 C. The muffle furnace is programmed for cool down to handling temperature. Transfer warm sand into 8 oz jars with minimal headspace. Wrap parafilm around the lids. Log into LIMS with a six month expiration date. Label and periodically run blanks to assure that it is interference free. Store away from interfering solvents.

7.1.14.2 Preparation of Reagent Sand option 2- Slowly add Ottawa sand into a one liter beaker half filled with reagent water while stirring. Let settle and decant the water to waste. Scoop wet sand into second beaker half filled with water while stirring. Repeat twice more. Spread wet sand out on a flat pan and dry in a drying oven over night or longer. Transfer warm sand into 8oz jars with minimal headspace. Wrap parafilm around the lids. Log into LIMS with a six month expiration date. Label and periodically run blanks to assure that it is interference free. Store away from interfering solvents.

7.1.14.3 Preparation of Reagent Sand option 3—Take a sample size of reagent sand and use 5 grams of the material as a blank. Analyze the sand the same as a reagent blank. If the sand is clean (all detection at least less than ½ the reporting limit) then the sand does not need further cleaning to be used for the method blank.

7.2 Standards

Stock Standard Solutions (SSS) – certified standards are purchased from approved vendors. The standards listed in this SOP are examples. SSS are used to prepare primary dilution standards (PDS), Working Standards (WS), and second source calibration verification standards (SCV). The source and composition of the SSS used to prepare a particular PDS, IS, or LCS is given in Section 17 Tables 3 & 4 of this SOP. Stock solutions are transferred from a flame sealed ampule to a 2 mL vial via a chilled Pasteur pipette, care must be taken avoid bubbling air through the liquid when transferring. Immediately cap the vial. The opening date is documented in LIMS and the expiration date is changed based on the guidelines provided in section 12.2.6 but are not to exceed the original manufacturer's expiration listed on the COA. Print a new label for the storage vial. The following SSS are used:

7.2.1 Target Analyte Mixes.

7.2.2 Surrogate Standard Mixes (SS).

7.2.3 Internal Standard Analyte Mixes (IS).

7.2.4 BFB Tuning Solution.

7.2.5 Labeled SSS solutions are stored in the freezer, refrigerator, or cabinet depending on manufacture's recommendations until they expire.

7.2.6 Frequency of Standard Preparation

Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases may need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented.

Dichlorodifluoromethane and chloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

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 one month for working standards and three 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

7.2.7 Original Certificates of Analysis (CofA's) of SSS are maintained in binders dedicated for the Certificates of Analysis and are available to personnel. CofA's may also be stored on servers that are available to personnel. A pdf of the CofA is attached to the standard in LIMS for easy reference. The certificates shall be used each time the method standards are prepared to confirm the concentration of analytes.

7.2.8 IS/SS are prepared as described in Section 17 Table 4 and immediately transferred to the OI 4100 standard vessel.

7.2.9 Labeled PDSs are stored in the freezer and replaced every 2-3 weeks or sooner when analytical results indicate a problem. Each Mininert vial containing the PDS shall be labeled in a standard format as described in section 9.1.5 including the expiration date.

7.3 Working Standards (WS) for water

Working Standards are prepared according to Section 17 Table 5. Working Standards are used for the initial calibration, initial and continuing calibration verification, LCS and LCSD.

7.3.1 MS's are prepared by taking a 49.95 mL aliquot of an environmental sample and spiking it with 50 µL of PDS.

7.3.2 The OI 4100 fills a 10 mL sampling loop via peristaltic pump, adds 2.0 ul of IS/SS PDS and transfers the aqueous solution to a 25ml purge vessel.

7.4 Working Standards for Medium Level Soils:

7.4.1 Working standard for medium level soils are prepared similarly to Section 17 Table 5 for ICAL, CCV, and SCV working standards except that a constant ratio of 1ml of purge & trap methanol to 50 mL of water is maintained in every standard. For example, CAL1 with the addition of 2mL of standard would include 998uL of blank methanol to 50mL of H2O and CAL7, 100 uL of PDS + 900 uL of methanol in 50 mL H2O.

7.4.2 A medium soil blank is prepared by weighing 5 +/-.04 g of reagent sand into 5 mL of methanol

7.4.3 LCSs for medium soils are spiked as follows. Create a 100ug/ml standard (100ul of the 4 Restek stocks listed in order at the top of Section 17 Table 3) + 1600 ul of methanol in a 2mL vial., Spike with 500 uL of the 100 ug/mL PDS and quickly add 4.5 mL of methanol. Invert no more than 3x. If methanol extracts were prepared in the field, weigh 5 g of reagent sand into retained methanol vial, remove 500 uL of methanol from the blank extract and then add the spike. This results in a 1000 ug/Kg spike.

7.4.4 Matrix spikes are prepared similarly to LCSs except for using sample instead of blank sand.

7.5 Working Standards (WS) for low level soils

7.5.1 Working Standards are prepared as per Section 17 Table 6. They are used for the instrument performance check, calibration, and calibration verification.

As per Section 17 Table 4, known amounts of methanolic PDS's are spiked directly to a 40 mL VOA bottle that contains 5.0 (±0.5 g) of Ottawa sand, 5 mL of 20% sodium bisulfate preservative and a magnetic stir bar.

7.5.2 All analysis samples and QC are automatically spiked with a consistent aliquot of IS/SS – usually between 1 and 5 uL by the autosampler directly to the sample while adding 10ml of reagent water resulting in a concentration of 50 µg/Kg. Concentration is based on the concentration in 5 g of soil. The addition of water does not affect the stated concentration of the standard or sample.

7.5.3 Retain records for all standards and reagents including the manufacturer/vendor, the Manufacture's Certificate of Analysis or purity, the date of receipt, recommended storage conditions, and an expiration date.

7.5.4 Document standards and reagents preparation in the LIMS System-indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date of prepared solution and preparer's initials.

7.5.5 Preservation of low level soils or aqueous samples can vary by project. Calibration for low level standards can be done without sodium bisulfite preservative as long as batch QC is preserved with the same amount and lot of preservatives in order to document their affect on recovery.

8.0 Sample Collection, Preservation, Shipment and Storage

PHILIS personnel do not take field samples, however proper samples containers and preservatives are listed in Section 17 Table 8.

PHILIS PT and QC samples should be treated exactly as actual samples. Use the date received as the sample date and allow LIMS to assign the standard hold times as per the method being analyzed. If the holding time is exceeded, document that it is a PT or QC sample that was received in a sealed ampoule and not opened until analysis. Once extracted or opened, normal holding times would be used and if times were exceeded, then the results would not be used.

8.1 Aqueous samples are collected in multiple 40 mL pre-cleaned VOA bottles.

8.1.1 Soil/Solid samples are collected in multiple 40 mL pre-cleaned VOA vials containing 5 mL of 20 % $NaHSO_4$ solution or 5 mL of methanol (for high level solids) and a magnetic stir bar.

8.1.2 Soil/Solid samples may also be taken in multiple Encore Samplers.

8.2 If there is a site specific sampling procedure as a part of the QAPP, then it automatically supersedes this SOP.

8.3 Samples are delivered to the PHILIS or appropriate field refrigerator for shipment to the lab for analysis within holding time.

8.4 The samples delivered to the PHILIS on the collection day must be transported in coolers containing ice to demonstrate the cooling process has begun. Samples shipped overnight to PHILIS must have temperatures that do not exceed 6 °C.

8.5 Samples are maintained at the temperature range from just above freezing to 6 °C

8.6 Temperature blanks should be included in the shipment with samples.

8.7 Trip blanks must be included with any volatile sample shipment.

8.8 If reagents are added in the field (e.g. 1:1 HCl), the same lot should be available to the lab for QC preparation.

9.0 Quality Control and Acceptance Criteria

QC requirements include the Demonstration of Capability and ongoing QC requirements that must be met when preparing and analyzing samples.

9.1 DEMONSTRATION OF CAPABILITY (DOC) – must be successfully performed by the analyst prior to analyzing any field samples and any time major method modifications are made. The following is done to demonstrate laboratory capability to perform this method:

9.1.1 Prior to conducting the DOC study, the analyst tunes the instrument, when required, and generates or verifies an acceptable instrument calibration following the procedure outlined in Section 10 of this SOP. A MB is analyzed to demonstrate that the background contamination is low enough to not interfere with analytes.

9.1.2 Method precision and accuracy are demonstrated by analyzing 4 replicate LCS's fortified at concentration listed in Section 17 Tables 5 or 6 and analyzed according to the procedure described in Section 11 of this SOP. Precision and accuracy limits are re-established every six months. Use current limits.

9.2 Ongoing QC applied when performing this method includes analyzing acceptable instrument calibration/calibration verification standards, method blanks, LCS, and MS/ MSD. Internal standards and surrogates must be acceptable with all QC samples and with test samples.

9.3 Lower limit of quantitation (LLOQ) – The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve. The verification is performed by the preparation and/or analysis of an LCS (or matrix spike) at 0.5 - 2 times the established LLOQ.

Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria of ±20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria. This practice acknowledges the potential for greater uncertainty at the low end of the calibration curve. Practical, historically based LLOQ acceptance criteria should be determined once sufficient data points have been acquired.

9.4 MDL Procedure

MDLs and RLs are established by analyzing a minimum of seven replicates of a standard at or near the estimated MDL. Tabulation of results and MDL calculations are performed by the method in 40 CFR, Part 136, Method Update Rule Revision 2.

9.4.1 Initial MDLs

9.4.1.1 Initial MDLs are established by analyzing a minimum of seven replicates of the low-level calibration standard and a minimum of seven blanks prepped and analyzed over three separate days. The MDL should be spiked 0.5 to 2 times the estimated MDL. Extract and analyze the MDL standards and blanks with the same procedure as regular samples.

9.4.1.2 For each compound, calculate the mean and standard deviation of the replicates in micrograms per liter (µg/L). Then calculate the MDL by multiplying the standard deviation by the Student's t value. The one-sided (single-tailed) Student's t values at the 99% confidence levels are used (e.g., t = 3.143 at the 99% confidence level for n = 7). MDL results are stored in Element each time they are calculated. This calculation must be performed separately for the spikes and blanks. If all blanks do not have analyte detection, then the largest blank value is taken for the blank MDL. If all blanks do have detection, use them to calculate the MDL with the formula above and add the mean of the blank result. If the mean of the blank results is a negative number then use 0 in place of the mean. The sum of these two numbers is the blank MDL. The larger of the MDL spike and MDL blank values is the MDL.

9.4.2 Ongoing MDL Data Collection

9.4.2.1 Ongoing MDL's are determined by preparing and analyzing two spiked standards at 1-5 times the estimated MDL and two blanks once per quarter for a minimum of seven determinations. The blanks and spikes may be analyzed in the same prep batch but is not required. If the instruments are being used regularly, the MDL spikes may be added to the routine batches and the regular blanks used. All blanks analyzed during the evaluation period should be used.

9.4.2.2 If no samples are analyzed during a quarter, then no ongoing MDLs are required. When this is the situation, MDLs, both spiked and blank, are analyzed with the PT samples analyzed every six months.

9.4.3 Annual calculations

At least once per year or a minimum of 13 months, evaluate the MDL by, calculating as above in 9.4.1.2. Use the larger of the spiked determinations and blank determinations for the mdl value. Include all data generated during the last twenty four months.

10.0 Calibration and Standardization

10.1 Prior to the analysis of samples, performance of the instrument is optimized, and an instrument calibration curve is developed. BFB is analyzed prior to instrument calibration, in order to verify that the mass abundance acceptance criteria specified in Section 17 Table 9 have been achieved. Tune checks are only required prior to ICAL, Alternatively, other published tuning criteria may be used provided that method performance is not adversely affected.

10.1.1 In order for data to be acceptable, all samples must be analyzed within a 12 hour window from the injection of the tune or CCV.

10.1.2 BFB (50 nanograms or less) can either be direct injected or purged into the system

10.1.2.1 Prepare a 50ug/ml standard in methanol by adding 1960ul of methanol to a 2mL screw cap vial. Add 40uL of a Restek Surrogate standard (Cat#30004) mix at 2500ug/mL.

For BFB injection using an autosampler, the maximum standard concentration shall be 50 µg/mL with a 1 µL injection. Using a purge and trap method, an aqueous standard at 10 µg/mL shall be used (inject 10 µL into a 50 mL vol flask), with a 10 mL purge to deliver 50 nanograms of BFB into the system. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan acquired within 20 scans of the elution of the BFB. Do not subtract part of the BFB peak or other discrete peak that does not coelute with the BFB. All calculations are performed by ChemStation Enviroquant or by MassHunter. BFB acceptance criteria are in Section 17 Table 9.

10.2 Setting Retention Times, Retention Time Windows and Integration Parameter

Once the purge and trap and GC cycle have finished for the midpoint or other calibration standard, load the quantitation file. Review each peak to make sure that the processing software identified the correct peak. If not, manually integrate the peak. Save all of the retention times. Quantify the calibration file and go through each ion profile of the target list, take note of the ion ratios, verify that the spectrum and profile match the standard spectrum.

10.3 The instrument is calibrated using standards at the seven (7) concentrations (given in Section 17 Tables 5, 6, or 7 based on the matrix) in the following manner: Cal1 – Cal 7 are used to generate the calibration curve for all target analytes. Average response factor or a linear regression curve can be used with five points, but a quadratic regression requires a minimum of six points. More or less standard points may be used provided required QA criteria can be met.

10.4 Rejection of calibration points

10.4.1 It is not generally acceptable to remove points from a calibration curve. Typically instrument maintenance and the accuracy of the calibration standards should be examined if the calibration acceptance criteria are not met.

10.4.2 If no problems are found, then a point(s) can be rejected as long as it meets the following criteria.

10.4.3 If the rejected point is the highest or lowest point in the ICAL, then the rejection may be done by analyte. If the rejected point is an internal point on the ICAL, it may be removed if the reason is obvious and the entire level (all analytes) is removed with the reason documented. Examples for level removal are; a bad injection, internal standards low or missing, contamination, etc.

10.5 The CAL1 standard contains the analytes at the RL of the analytical method. The lowest concentration included in the calibration curve is the reporting limit.

10.6 A response factor calibration curve is generated for each target analyte by plotting the response factor as a function of concentration ratio. If the analyte does not meet the 20% variability acceptance criteria, then a regression fit should be used. If linear or quadratic regression is used, the resulting curve fit must be 0.99 or greater.

10.7 All calibration points must be recalculated (not reanalyzed) using the final calibration curve in which this standard is used (i.e., re-fitting the response from the calibration standard back into the curve). The recalculated concentration of the calibration standard corresponding to the LLOQ must be within ±50% of the standard's true concentration and within ±30% for all others (i.e. above the low standard).

10.8 Recommended initial and continuing calibration relative response factors (RRFs) for the volatile compounds are listed in Section 17 Table 10. If the response is below the recommended limit, then the calibration must be evaluated to determine that the lowest point on the calibration curve may be distinguished from the baseline.

10.9 The instrument calibration curve is initially verified by the SCV and continuously verified every 12 hours by the CCV. The concentration of the CCV is ≤ the midlevel of the calibration curve.

10.10 The Relative Error of the calibration curve is determined by processing the lowest point and the midpoint of the calibration against the curve. The % difference in true value is the Relative Error.

10.11 Acceptance criteria for the Instrument Calibration and CCVs, and the required frequency of their analysis are summarized in Section 17 Table 11.

11.0 Procedure

Samples can be prepared after passing an SVC, after an ICAL or CCV from the same matrix as the samples. An instrument blank may be run after a CCV or LCS if carryover at the CCV level is anticipated.

11.1 Sample Preparation for Aqueous Samples based on SW846 Method 5030C

11.1.1 Remove samples from the laboratory refrigerator and allow to equilibrate to ambient temperature.

11.1.2 Prepare and analyze the batch blank and LCS from with the same amount and lot of reagents and preservatives as the samples.

11.1.3 Verify that they have been logged into the LIMS and are within the holding time. If the sample exceeds holding time notify the Lead Chemist and follow the corrective action plan.

11.1.4 Transfer the internal custody of the container being analyze to APL01 or PAL and mark as Active Out from sample receiving or the refrigerator where sample are stored. Verify that you are logged into your own LIMS account.

11.1.5 Ensure that the 40 mL sampling bottles are free of headspace. If headspace is noticed, notify the Lead Chemist and follow the corrective action plan. Headspace is considered a problem is the bubble is greater than ¼ inch in diameter (the size of a pea).

11.1.6 Analyze no more than 20 samples per batch.

11.1.7 Load vials on the sampling tray and run the water program on the auto sampler. Verify that the correct standard addition vessel is being used. 10ml of the water samples will be added to the sparge vessel while adding 2ul of the internal standard surrogate solution

11.1.8 Once analyzed, the pH and total and residual chlorine level of the spent container can be determined by test strip and recorded into the special info section of the batch bench sheet.

11.1.9 For samples to be analyzed as MS/MSD follow the procedure below:

11.1.9.1 The client must provide four (4) additional samples to be analyzed as MS/MSD in addition to the original 40 mL VOA bottle.

11.1.9.2 To create an MS sample, transfer approximately 44 mL of the sample to a 50 mL volumetric flask.

11.1.9.3 Spike 50 µL of LCS PDS to the volumetric flask and dilute to the 50 mL mark with the sample only, not reagent water.

11.1.9.4 For MSD samples, repeat the procedure for the MS sample

11.2 Sample preparation for Medium Level Soil Samples based on SW846 Method 5035A.

11.2.1 Verify the calibration of a top loading using certified weight which cover the range of the samples to be weighed. Check off the serial number of the weights and balance in the sample weight log look. The top loading balance in APL01 and SPA01 read to the hundredth of a gram. It is a NELAP requirement that sample weights for 8260D and calibration weights be recorded to the same number of significant figures. Record the calibration weights accordingly.

11.2.2 Remove the sample samples from the refrigerator or sample receiving and transfer the internal custody of the containers removed appropriately.

11.2.3 Samples from 5g Encore samples must be extracted or frozen on the day of receipt. Tare a 40ml vial on a top loading balance. Extrude the contents of the Encore sampler into the vial and. Record the weight in the logbook. Add 5ml of purge and trap methanol and cap the vial. Allow the methanol to moisten the core and gently shake to break up the core and/or mix the vial contents. Agitate the extracts periodically over the course a half an hour. vials Allow to settle until methanol on top is clear. If the sample does not settle samples may be loaded into a centrifuge (balance the load when adding to a centrifuge) and spin briefly at a low setting to settle out the contents

11.2.4 Samples preserved with methanol in the field are prepared by recording the tare weight determining the final weight and logging into a sample weight log.

11.2.5 Batch QC is by adding weighing 5+/- 0.04g of reagent sand to the 5ml methanol in 40ml vials that were retained by the lab when preparing sample kits or adding 5ml methanol to 5+/- 0.04g of reagent sand in the lab.

11.2.6 The LCS blank extract is spiked as follows: Create a 100ug/ml standard (100ul of the 4 Restek stocks listed in order at the top of Section 17 Table 3) + 1600ul of methanol in a 2ml vial. Remove 500ul of methanol from the extract. Spike with 500ul of the 100ug/ml PDS. Invert no more than 3x.

11.2.7 Final weights are all recorded in the batch bench sheet,

11.2.8 Methanol extracts can be screened by headspace prior to analyses. See SOP-L-A-102 for this procedure.

11.2.9 Extracts are analyzed at a default dilution of x50 by transferring 1.0ml of the sample or QC extract via gas tight syringe to a 50ml volumetric flask containing approximately 48ml of reagent water and then bring up to the mark with reagent water via pasteur pipette. If a dilution is being run with a lesser aliquot of extract add additional methanol to bring the total volume of methanol in 50mL of water to 1.0 mL.

11.2.10 Load vials on the sampling tray and run the water program on the OI 4100. Verify that the correct standard addition vessel is being used. 10ml of the dilutions will be added to the sparge vessel while adding 2uL of the internal standard surrogate solution.

11.3 Preparation and analysis of Low Level Soil Samples

11.3.1 Remove the samples from the refrigerator or sample receiving.

11.3.2 Verify that they have been logged into LIMS and are within holding time. If the sample exceeds holding time, notify the Lead Chemist and follow the corrective action plan.

11.3.3 Edit the internal custody log in LIMS for the containers that you are working with.

11.3.4 Batch no more than 20 samples to be analyzed.

11.3.5 Reweigh the samples and ensure the original tare weight was recorded. If it was lost, damaged or destroyed, notify the Lead Chemist and follow the corrective action plan.

11.3.6 Weigh the samples, record the weights in the sample weight log Determine the final weight and log into the batch bench sheet in LIMS.

11.3.7 The contents of Encore samples are extruded into a tared 40ml vial. Weights are recorded and 5ml of reagent water or 5ml of 20% Sodium bisulfate are added depending on when the samples are to analyzed and or the QAPP. Also add a magnetic stir bar.

11.3.8 Batch blanks are prepared in retained vials with water or sodium bisulfate if the samples were added in the field.5.0 +/- .04 g of reagent sand are weighed int the vial.

11.3.9 Batch blanks for Encore samples prepared in the lab. 5. +/- -0.04g of reagent sand is weighed int a 40ml tared vial, a stir bar and 5 ml of reagent water or 20% sodium bisulfate solution from the sample preparation lot is added along with a magnetic stir bar.

11.3.10 LCS and matrix spikes are spiked with a known concentration of PDS directly into the prepared sample or QC sample and swirled to mix in the spike.

11.3.11 Sample and QC are loaded on the auto sampler tray. A soil program is chosen for analysis after verifying the correct standard vessel is being used.

11.3.12 Fresh reagent water is added to the instrument reservoir.

11.3.13 10ml of water is added to vial while adding 1-5 uL of a known concentration of internal standard/surrogate mix. The sample is preheated to 40C and purged through a multistage needle while stirring.

11.4 Standard Preparation

Follow the procedure listed in Section 17 Table 5 for waters, Section 17 Table 6 for low level soils, and Section 17 Table 7 for high level soils.

11.5 Sample Analysis- Sections 11,1, 11,2, and 11.3 cover the loading of the OI 4100 for various matrices. Example Purge and Trap and GC/MS acquisition parameter

11.5.1 In ChemStation, load the "default" or previous day's sequence. Make sure the sequence ties the correct method and tune settings for the samples being analyzed. Make the changes as necessary to reflect the QC and samples that you will be analyzing. A typical sequence would start with the tune (only required if calibrating), CCV, MB, and LCS. It would then contain samples and an MS/MSD or MS/Sample Dup. Save the sequence with the instrument ID and the date of analysis. Instrument blanks after standards or LCS sample if carryover contamination is anticipated.

11.5.2 Check the helium supply, water supply, and IS/SS to make sure an adequate amount is available to complete the sequence.

11.5.3 Start the sequence.

11.6 Identification of Analytes

11.6.1 The analyte is identified by comparison of its mass spectrum to a reference spectrum updated in the instrument method. The major ions (greater than 10% of the most abundant ion should be present in the spectra. The relative % of the major ions should be within ±20% of the expected abundance.

11.6.2 The analyte is also identified by its retention time compared to the retention time observed for the same analyte in the most recent retention time update. The retention time must be within 10 seconds of the midpoint of the calibration curve or most recent CCV for that compound. An RRT value may also be used. If retention time criteria fails and judgement from qualitative MS data indicate a positive hit notify the lead chemist to see if the data should be reported with a flag.

11.7 Quantitation of Analytes

Analytes are quantified by comparing the response to that in the calibration curve and multiplied by any dilution or sample preparation factor. A list of the analytes and quant ions is listed in Section 17 Table 13.

12.0 Data Analysis and Calculations

12.1 The concentration of each analyte is calculated using Agilent MSD ChemStation software using an average response factor or linear regression curve as established in Section 10 of this SOP. Response factors and analyte concentrations are calculated by the equations below:

12.2 Relative response factor (RRF):

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

where:
A_x = Area of the quantitation ion for the surrogate or compound being measured.
A_{is} = Area of the quantitation ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the surrogate or compound being measured.

12.3 Average RRF:

$$\overline{RRF} = \frac{\sum_{1}^{n} RRF}{n}$$

where
n = number of initial calibration standards

12.4 Percent relative standard deviation (%RSD):

$$\%RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:
s = standard deviation: $s = \sqrt{\frac{\left(\sum_{i=0}^{n}(\bar{x} - x_i)^2\right)}{n-1}}$

12.5 $\bar{x} = \overline{RRF}$:

$$\overline{RRF} = \frac{\sum_{1}^{n} RRF}{n}$$

12.6 Sample concentration using RRF:

$$\text{Conc.}\left(\frac{\mu g}{L}\right) = \frac{(A_x)(I_s)(D)}{(\overline{RRF})(V_o)(A_{IS})}$$

where :
A_x = area of quantitation ion for compound being measured
I_s = amount of internal standard injected (ng)
A_{is} = area of quantitation ion for the internal standard
$\overline{RRF}$ = mean relative response factor for compound being measured
V_o = volume of water extracted purged (mL) accounting for dilutions
D = Dilution Factor

12.7 Percent recovery for CCV, ICV, LCS, and MS are performed using the following equation:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:
C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample; (Cx = 0 for CCV, LCS and ICV.)
C_t = the theoretical spike concentration.

12.8 Relative percent difference for duplicate is calculated using the following equation:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:
C_1 = concentration of the first sample
C_2 = concentration of the second sample

12.9 Instrument generated data goes through a series of reviews prior to being submitted to the client. First the analyst reviews the data to ensure method and client requirements are met. Then the instrument data goes through a peer review covering the same items as the analyst. Both reviews are documented on Form QA-017, which is provided in Figure 2. The Quality Assurance Manager also reviews a minimum of 10 % of data to evaluate the QA process.

12.10 Analytical data generated by the instrument software is reviewed and evaluated by the analyst and peer as follows:

12.10.1 BFB, instrument calibration, calibration verifications, IS/SS, QC measures are evaluated and the results documented on the separate forms.

12.10.2 The tune evaluation of BFB.

12.10.3 The instrument calibration relative response factors and percent relative standard deviations.

12.10.4 QA-QC check report for internal standard area counts and percent recoveries for the surrogates.

12.10.5 Analyte percent recoveries CCV, LCS, SCV, MS, and RPD for MSD.

12.11 In order for the analytical data to be acceptable, the calibration standards and quality control measures must meet the criteria listed in Sections 9 and 10 of this SOP including Section 17 Tables 9 - 11 and 14.

12.12 All false positives are Q-Deleted, and all positively identified target analytes are reported to LIMS.

12.13 Manual integration is applied in cases when the instrument data processing software produces integrated areas that are not valid. The manual adjustments to the chromatographic peak must be performed in a consistent manner for the calibration standards, QC and field samples.

Manual integration should not be substituted for proper maintenance of the instrument or setup of the method (e.g. retention time updates), integration parameter files, etc.

The analyst should seek to minimize manual integrations by proper instrument maintenance, retention time updates, and configuring peak integration parameters.

12.14 If the QAPP requires it, chromatograms of all field samples are examined to detect additional peaks, which were not identified as target analytes. If such peaks are present, generate a Library Search Report and report a tentatively identified compound (TIC) if the percent match is greater than the 50%. The Lead Chemist should be notified

immediately in that case. An example chromatogram is shown in Figure 1. Method 8260D Revision 4 Section 11.6.2 provides guidelines for tentative identification of non target peaks.

12.15 Anytime the analyst alters the instrument generated quantitation report, the hardcopies of both reports (original and analyst's corrected) must be retained (e.g., manual integration). The altered report must be initialed and dated by the analyst with a reason for altering. The corrected report must also be reviewed, initialed, and dated by a peer or supervisor.

12.15.1 Discrepancies in the analytical run are documented on the "Data Review Form, QA-017" and discussed with the Lead Chemist. "Data Review Form QA-017" is signed by the Level 1 reviewer, the Level II reviewer, and Quality Assurance.

12.16 Reviewed data is entered into LIMS, hard copies of LIMS report is printed and compared to the original data or may be reviewed in LIMS.

12.17 All records derived from the analytical process are assembled in the analytical data packages that consist of:

12.17.1 Analytical run sheet.

12.17.2 BFB tune evaluation report, if analyzed.

12.17.3 QA-QC check report.

12.17.4 Quantitation Report for each Sample and QCS.

12.17.5 Evaluation reports for CCV, SCV, LCS, MS, and MSD.

12.17.6 Initial calibration form.

12.18 Data packages are maintained electronically on servers in multiple locations.

12.19 Corrective Actions for Out of Control

In cases where quality control measures do not meet acceptance criteria, the quality of the analytical data is not acceptable, and the analyst does the following:

12.19.1 When tuning and instrument calibration fail to meet acceptance criteria, the analysis does not start. The problem is investigated, and the necessary instrument maintenance is performed, followed with tuning and calibration. Instrument tuning is only required prior to calibration.

12.19.2 If the acceptance criteria listed in Section 17 Tables 11 & 14 of this SOP are not met for ICAL, SCV, CCV, MB, LCS, MS, MSD, internal standards, and surrogates, then the affected QCs and associated samples should be treated as per laboratory or QAPP protocols.

12.19.3 If after analysis, any of the criteria for quality control are not met, or the sample is not available for reanalysis, the analyst must notify the Lead Chemist. The Lead Chemist will implement the corrective action plan.

12.20 The analyst shall report to the Lead Chemist and indicate on the "Data Review Form QA-017" any out of control event. Such events include:

12.20.1 Damage to the sample.

12.20.2 Headspace in the sample bottle.

12.20.3 Holding time exceeded.

12.20.4 Inadequate sample preservation.

12.20.5 Sample results exceeds the Agency's action limit

12.20.6 Samples do not reflect historical data.

12.20.7 Upward trending or sample results approaching interval warning limits.

12.20.8 Any non-target analyte peak present on the instrument generated chromatogram that interferes with target analyte peaks.

12.21 The Lead Chemist will implement the corrective action plan described in the PHILIS corrective action plan document.

12.22 See the QAPP that the samples were analyzed under for contingencies or guidance on handling out of control or unacceptable data.

13.0 Method Performance

13.1 Demonstration MDL data is presented in Section 17 Tables 1 & 2. MDLs are analyzed at least annually or with instrumentation changes. Lab Accuracy and Precision data are used to calculate lab specific acceptance criteria. Precision and Accuracy data are recalculated and evaluated every six months. Limit acceptance criteria will be established no tighter than 80 % to 120 % for accuracy and 20% for precision.

13.2 Other specific Quality Assurance Objectives (QAO) may be found in the appropriate statement-of-work or Quality Assurance Project Plan (QAPP) for specific projects.

14.0 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. The EPA places pollution prevention as the management option of first choice with regard to laboratory waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The Environmental Protection Agency requires that laboratory waste management practices be compliant with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult the PHILIS Chemical Hygiene Plan.

14.3 The waste produced from EPA Method 5030C or 5035A/8260D consist of waste collected from the purge and trap system, excess sample, standards (stock mixes, PDS, WS), and methanol.

14.4 Waste from the purge and trap system from field samples are disposed in the Hazardous Waste container.

14.5 Excess reagents are disposed following the MSDS instructions.

14.6 Glass pipettes are disposed in the glassware waste.

14.7 Refer to EPA Method 8260D, section 14.0 and 15.0 for additional guidance.

14.8 For information about pollution prevention that may be applicable to laboratories and research institutions consult Less is Better: Laboratory Chemical Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036.

15.0 Waste Management

Waste management procedures are specified in the Hazardous Waste Management Plan.

16.0 References

16.1 EPA Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Revision 4, June 2018; U.S. EPA Office of Solid Waste.

16.2 EPA Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Revision 3, August 2006; U.S. EPA Office of Solid Waste.

16.3 EPA Method 5030C, Purge-And-Trap of Aqueous Samples, Revision 3, May 2003; U.S. EPA Office of Solid Waste.

16.4 EPA Method 5035A, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July 2002.

16.5 2003, 2009, and 2016 NELAP manuals.

16.6 40 CFR 136, Appendix B, Revision 2. Definition and Procedure for the Determination of the Method Detection Limit – December, 2016

16.7 U.S. EPA Contract Laboratory Program Statement of Work OLM 04.2.

16.8 U.S. EPA National Functional Guidelines, Superfund Organic Methods Data Review, June, 2008.

17.0 Tables, Diagrams, Flowcharts and Validation Data

Table 1. Examples of Analytes, MDLs, and RLs for Water using EPA Method 8260D

Analyte	CAS #	MDL (µg/L)	RL (µg/L)
Dichlorodifluoromethane	75-71-8	0.3	5.0
Chloromethane	74-87-3	0.6	5.0
Vinyl Chloride	75-01-4	0.7	5.0
Bromomethane	74-83-9	1.1	5.0
Chloroethane	75-00-3	0.8	5.0
Trichlorofluoromethane	75-69-4	0.5	5.0
1,1-Dichloroethene	75-35-4	1.4	5.0
t-Butyl alcohol	75-65-0	10.1	25
Carbon disulfide	75-15-0	0.7	5.0
Iodomethane	74-88-4	0.4	5.0
Methylene chloride	75-09-2	0.6	10.0
Methyl-tert-Butyl ether	1634-04-4	0.3	5.0
Acetone	67-64-1	8.4	25.0
trans-1,2-Dichloroethene	156-60-5	1.4	5.0
Di isiopropyl ether	108-20-3	0.3	5.0
1,1-Dichloroethane	75-34-3	0.5	5.0
cis-1,2-Dichloroethene	156-59-2	0.6	5.0
2,2-Dichloropropane	594-20-7	2.0	10.0
Bromochloromethane	74-97-5	0.7	5.0
Chloroform	67-66-3	0.6	5.0
Carbon tetrachloride	56-23-5	1.0	5.0
1,1,1-Trichloroethane	71-55-6	1.7	5.0
t-Amyl methyl ether	994-05-8	.3	5.0
1,1-Dichloropropene	563-58-6	0.5	5.0
2-Butanone	78-93-3	4.7	25.0
Ethyl tert-butyl ether	637-92-3	.2	5.0
Benzene	71-43-2	0.4	1.0
1,2-Dichloroethane	107-06-2	0.5	5.0
Trichloroethene	79-01-6	0.6	5.0
Dibromomethane	74-95-3	0.6	5.0
1,2-Dichloropropane	78-87-5	0.5	5.0
Bromodichloromethane	75-27-4	0.5	5.0
cis-1,3-Dichloropropene	10061-01-5	1.0	5.0
Toluene	108-88-3	0.5	5.0
Tetrachloroethene	127-18-4	0.3	5.0

Analyte	CAS #	MDL (µg/L)	RL (µg/L)
trans-1,3-Dichloropropene	10061-02-6	0.8	5.0
1,1,2-Trichloroethane	79-00-5	0.6	5.0
Dibromochloromethane	124-48-1	0.3	10.0
1,3-Dichloropropane	142-28-9	0.6	5.0
1,2-Dibromoethane	106-93-4	0.8	5.0
2-Hexanone	591-78-6	7.1	25.0
Chlorobenzene	108-90-7	0.6	5.0
Ethyl benzene	100-41-4	0.3	5.0
1,1,1,2-Tetrachloroethane	630-20-6	0.5	5.0
m,p-Xylenes	1330-20-7	0.4	10.0
o-Xylene	95-47-6	0.5	5.0
Bromoform	75-25-2	0.4	5.0
Styrene	100-42-5	0.4	5.0
Isopropylbenzene	98-82-8	0.3	5.0
Bromobenzene	108-86-1	0.5	5.0
n-Propylbenzene	103-65-1	0.4	5.0
1,1,2,2-Tetrachloroethane	79-34-5	1.0	5.0
2-Chlorotoluene	95-49-8	0.3	5.0
1,2,3-Trichloropropane	96-18-4	0.9	5.0
1,3,5-Trimethylbenzene	108-67-8	0.3	5.0
4-Chlorotoluene	106-43-4	0.4	5.0
tert-Butylbenzene	98-06-6	0.3	5.0
1,2,4-Trimethylbenzene	95-63-6	0.4	5.0
sec-Butylbenzene	135-98-8	0.3	5.0
1,3-Dichlorobenzene	541-73-1	0.5	5.0
p-Isopropyltoluene	99-87-6	0.4	5.0
Butylbenzene	104-51-8	0.4	5.0
1,4-Dichlorobenzene	106-46-7	0.5	5.0
1,2-Dichlorobenzene	95-50-1	0.7	5.0
1,2-Dibromo-3-chloropropane	96-12-8	0.6	5.0
1,2,4-Trichlorobenzene	120-82-1	1.0	5.0
Hexachlorobutadiene	87-68-3	0.8	5.0
Naphthalene	91-20-3	1.1	5.0
1,2,3-Trichlorobenzene	87-61-6	1.0	5.0
4-Methyl-2-Pentanone	108-10-1	7.0	25.0

Table 2. Examples of Analytes, MDLs, and RLs for Soils using EPA Method 8260D

Analyte	CAS #	MDL (µg/kg)	RL (µg/kg)
Dichlorodifluoromethane	75-71-8	2.2	5.0
Chloromethane	74-87-3	1.0	5.0
Vinyl Chloride	75-01-4	1.2	5.0
Bromomethane	74-83-9	1.0	5.0
Chloroethane	75-00-3	1.0	5.0
Trichlorofluoromethane	75-69-4	2.3	5.0
1,1-Dichloroethene	75-35-4	2.0	5.0
t-Butyl alcohol	75-65-0	9.5	20.0
Carbon disulfide	75-15-0	1.7	5.0
Iodomethane	74-88-4	1.2	5.0
Methylene chloride	75-09-2	2.1	20.0
Methyl-tert-Butyl ether	1634-04-4	1.0	5.0
Acetone	67-64-1	16.3	20.0
trans-1,2-Dichloroethene	156-60-5	1.7	5.0
Di isiopropyl ether	108-20-3	0.8	5.0
1,1-Dichloroethane	75-34-3	0.8	5.0
cis-1,2-Dichloroethene	156-59-2	0.7	5.0
2,2-Dichloropropane	594-20-7	1.8	5.0
Bromochloromethane	74-97-5	0.7	5.0
Chloroform	67-66-3	0.7	5.0
Carbon tetrachloride	56-23-5	1.7	5.0
1,1,1-Trichloroethane	71-55-6	1.2	5.0
t-Amyl methyl ether	994-05-8	2.0	5.0
1,1-Dichloropropene	563-58-6	2.7	5.0
2-Butanone	78-93-3	2.4	10.0
Ethyl tert-butyl ether	637-92-3	1.6	5.0
Benzene	71-43-2	1.1	5.0
1,2-Dichloroethane	107-06-2	0.7	5.0
Trichloroethene	79-01-6	2.4	5.0
Dibromomethane	74-95-3	0.7	5.0
1,2-Dichloropropane	78-87-5	0.8	5.0
Bromodichloromethane	75-27-4	0.7	5.0
cis-1,3-Dichloropropene	10061-01-5	1.1	5.0
Toluene	108-88-3	1.9	5.0
Tetrachloroethene	127-18-4	4.7	10.0

Analyte	CAS #	MDL (µg/kg)	RL (µg/kg)
trans-1,3-Dichloropropene	10061-02-6	0.7	5.0
1,1,2-Trichloroethane	79-00-5	0.6	5.0
Dibromochloromethane	124-48-1	0.7	5.0
1,3-Dichloropropane	142-28-9	0.8	5.0
1,2-Dibromoethane	106-93-4	0.8	5.0
2-Hexanone	591-78-6	5.0	10.0
Chlorobenzene	108-90-7	1.8	5.0
Ethyl benzene	100-41-4	2.4	5.0
1,1,1,2-Tetrachloroethane	630-20-6	0.8	5.0
m,p-Xylenes	1330-20-7	2.7	5.0
o-Xylene	95-47-6	2.0	5.0
Bromoform	75-25-2	1.0	5.0
Styrene	100-42-5	2.6	5.0
Isopropylbenzene	98-82-8	2.6	5.0
Bromobenzene	108-86-1	1.7	5.0
n-Propylbenzene	103-65-1	3.1	5.0
1,1,2,2-Tetrachloroethane	79-34-5	2.1	5.0
2-Chlorotoluene	95-49-8	2.6	5.0
1,2,3-Trichloropropane	96-18-4	1.6	5.0
1,3,5-Trimethylbenzene	108-67-8	3.1	5.0
4-Chlorotoluene	106-43-4	2.9	5.0
tert-Butylbenzene	98-06-6	2.4	5.0
1,2,4-Trimethylbenzene	95-63-6	3.3	5.0
sec-Butylbenzene	135-98-8	3.1	5.0
1,3-Dichlorobenzene	541-73-1	2.6	5.0
p-Isopropyltoluene	99-87-6	3.1	5.0
Butylbenzene	104-51-8	3.3	5.0
1,4-Dichlorobenzene	106-46-7	2.7	5.0
1,2-Dichlorobenzene	95-50-1	2.1	5.0
1,2-Dibromo-3-chloropropane	96-12-8	1.6	5.0
1,2,4-Trichlorobenzene	120-82-1	3.0	5.0
Hexachlorobutadiene	87-68-3	2.7	5.0
Naphthalene	91-20-3	1.5	5.0
1,2,3-Trichlorobenzene	87-61-6	2.4	5.0
4-Methyl-2-Pentanone	108-10-1	4.6	10.0

Table 3. Example Preparation of PDS, IS/SS, SCV-PDS in Methanol for Analyses of Aqueous Samples and Methanol Extractions

PDS Name	SSS Mix Used				Solvent	PDS description		
	Source/catalog#	Analytes	Conc. µg/mL	Volume µL	Volume mL	Conc. µg/mL	Final Volume mL	Application
PDS	Restek/30633	Mega Mix	2,000	500	MeOH 1.8	50	2.00	ICS, CCV, MS, MSD, LCS, LCSD
	Restek/30042	Gases	2,000	50		50		
	Restek/30006	Ketones	5,000	50		125		
	Restek/30465	Oxygenates	2,000	50		50		
IS/SS	Restek/30241	IS	2,500	1,000	MeOH	250	10.00	All Samples & Standards
	Restek/30004	SS	2,500	1,000	8.0	250		
SCV-PDS	AccuStandard/ M-502B-10X	Gases	2000	50	MeOH 1.725	50	2.00	SCV
	AccuStandard/ M-502A-R-10X	Liquids	2000	50		50		
	Accustandard CLP-22K-10X	Ketones	2000	125		125		
	Accustandard OGAD-001	Oxygenates	2000	50		50		

Note: tert-Butyl alcohol is 5x, ketones are 2.5x and m+p-Xylene is 2x the concentration stated in all preparations.

Table 4. Example Preparation of PDS, IS/SS, SCV-PDS methanol standards for Analyses of Soil/Solids

PDS Name	SSS Mix Used				Solvent	PDS description		
	Source/catalog#	Analytes	Conc. µg/mL	Volume µL	Volume mL	Conc. µg/mL	Final Volume mL	Application
PDS 50	Restek/30633	Mega Mix	2,000	50	MeOH 1.8	50	2.00	ICS, CCV, MS, MSD, LCS, LCSD
	Restek/30042	Gases	2,000	50		50		
	Restek/30006	Ketones	5,000	50		125		
	Restek/30465	Oxygenates	2,000	50		50		
PDS 5	PDS 50	All above.	50	200	MeOH 1.8	5	2.00	CAL/MRL
IS/SS	Restek/30241	IS	2,500	500	MeOH	125	10.00	All Samples & Standards
	Restek/30004	SS	2,500	500	8.0	125		
SCV-PDS	AccuStandard/ M-502B-10X	Gases	2000	50	MeOH 1.725	50	2.00	SCV
	AccuStandard/ M-502A-R-10X	Liquids	2000	50		50		
	Accustandard CLP-22K-10X	Ketones	2000	125		125		
	Accustandard OGAD-001	Oxygenates	2000	50		50		

Note: tert-Butyl alcohol is 5x, ketones are 2.5x and m+p-Xylene is 2x the concentration stated in all preparations.

Table 5. Example Preparation of Aqueous Working Standards in Edison

Working Standard Name	WS Conc. (µg/L) Analytes	Vol (µL)		Final Volume Water (mL)
		PDS	SCV-PDS	
Cal 1	2.0	2.0	-	50.00
Cal 2	5.0	5.0	-	50.00
Cal 3	10.0	10.0	-	50.00
Cal 4	20.0	20.0	-	50.00
Cal 5	50.0	50.0	-	50.00
Cal 6	80.0	80.0	-	50.00
Cal 7	100.0	100.0	-	50.00
CCV	20.	20.		50.00
SCV	50.	-	50	50.00
LCS	20.	20	-	50.00

Note: tert-Butyl alcohol is 5x, ketones are 2.5x and m+p-Xylene is 2x the concentration stated in all preparations.

Table 6. Example Preparation of Working Standards for Analyses of Low Level Soil/Solids

Working Standard Name	WS Conc. (µg/kg) Analytes	Vol (µL)			Mass Sand (g)	Vol 20% $NaHSO_4$ (mL)
		PDS-5	PDS-50	SCV-PDS		
Cal 1	5	5	-	-	5.0	5.0
Cal 2	10	10	-	-	5.0	5.0
Cal 3	20	20	-	-	5.0	5.0
Cal 4	50	-	5	-	5.0	5.0
Cal 5	80	-	8	-	5.0	5.0
Cal 6	100	-	10	-	5.0	5.0
Cal 7	150	-	15	-	5.0	5.0
CCV	50	-	5	-	5.0	5.0
SCV	50	-	-	5	5.0	5.0
LCS	50	-	5	-	5.0	5.0

Note: tert-Butyl alcohol is 5x, ketones are 2.5x and m+p-Xylene is 2x the concentration stated in all preparations.

Table 7. Example Preparation of Aqueous Working Standards for Analyses of High Level Soils

Working Standard Name	WS Conc. (µg/kg) Analytes	Vol (µL)		Final Volume Water (mL)
		PDS	SCV-PDS	
Cal 1	100.	2.0	-	50.00
Cal 2	250.	5.0	-	50.00
Cal 3	500.	10.0	-	50.00
Cal 4	1000	20.0	-	50.00
Cal 5	2500	50.0	-	50.00
Cal 6	4000	80.0	-	50.00
Cal 7	5000	100.0	-	50.00
CCV	1000	20.0	-	50.00
SCV	2500	-	50	50.00
LCS	1000	20.0		50.00

Note: tert-Butyl alcohol is 5x, ketones are 2.5x and m+p-Xylene is 2x the concentration stated in all preparations.

Table 8. Recommended VOC Sample Preservation Techniques and Holding Times taken from SW 846 Method 5035A and Chapter 4 Table 4-1

Sample Matrix	Preservative	Holding Time	Comment
Aqueous Samples No Residual Chlorine Present	Cool to 4 ± 2° C.≤ 6 °C	7 days	If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples No Residual Chlorine Present	Cool to ≤ 6 °C and adjust pH to less than 2 with HCl or solid NaHSO4.	14 days[1]	Reactive compounds such as 2-chloroethylvinyl either readily break down under acidic conditions. If these types of compounds are analyst of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
Aqueous Samples Residual Chlorine Present	Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to ≤ 6 °C.	7 days	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples Residual Chlorine Present	Collect sample in a pre-preserved container containing e 25 mg ascorbic acid per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to ≤ 6 °C and adjust pH to less than 2 with HCl	14 days[1]	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. Caution: never add acid preservative directly to a dechlorinating agent prior to sample collection.
Solid Samples[2]	Sample is extruded into an empty sealed vial and frozen on-site to < -7° C.	14 days[1]	Sample vials should not be frozen below -20° C due to potential problems with vial seals and the loss of constituents upon sample thawing.
Solid Samples[2]	Sample is extruded into an empty sealed vial and cooled to ≤ 6 °C for no more than 48 hours then frozen to < -7° C upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20° C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into an empty sealed vial and cooled to ≤ 6 °C for no more than 48 hours then preserved with methanol upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not preserved with methanol prior to the expiration of the 48 hour period.

Sample Matrix	Preservative	Holding Time	Comment
Solid Samples[2]	Sample is extruded into an empty sealed vial and cooled to ≤ 6 °C.	48 hours	
	Cool to ≤ 6 °C the coring tool used as a transport device.	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7° C or chemically preserved. Coring tools should not be frozen below -20° C due to the potential problems with tool seals and the loss of constituents upon sample thawing.
	Freeze to < -7° C the coring tool used as a transport device	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to < -7° C or chemically preserved. Coring tools should not be frozen below -20° C due to the potential problems with tool seals and the loss of constituents upon sample thawing.
Solid Samples[2]	Sample is extruded into a vial containing reagent water and frozen on-site to < -7° C.	14 days[1]	Sample vials should not be frozen below -20° C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into a vial containing reagent water and cooled to ≤ 6 °C for 48 hours or less then frozen to < -7° C upon laboratory receipt.	14 days[1]	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20° C due to potential problems with vial seals and the loss of constituents upon sample thawing.
Solid Samples[2]	Sample is extruded into a vial containing reagent water and 1 g $NaHSO_4$ and cooled to ≤ 6 °C.	14 days[1]	Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
	Sample is extruded into a vial containing methanol and cooled to ≤ 6 °C.	14 days[1]	Additional methanol extract storage time beyond 14 days may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

Notes:

1 A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

2 For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

Table 9. BFB Relative Abundance Suggested Criteria

* BFB Relative Abundance Criteria	
m/z	**Relative Abundance Criteria**
95	50 to 200% of mass 174
96	5 to 9% of 95 with Helium carrier gas
173	<2% of 174
174	50-200% or mass of 95
175	5 to 9% of 174
176	95 to 105% of m/z 174
177	5 to 10% of m/z 176

Note: * Criteria based on EPA Method 524.4 (Reference 17), with modified m/z 95 and m/z 174 abundance criteria.

Table 10. Example Relative Response Factor Criteria for Initial and Continuing Calibration Verification

Compounds	Minimum Response Factor	Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100	1,2-Dichloropropane	0.100
Chloromethane	0.100	Bromodichloromethane	0.200
Vinyl Chloride	0.100	Cis-1,3-Dichloropropene	0.200
Bromomethane	0.100	Trans-1,3-Dichloropropene	0.100
Chloroethane	0.100	4-Methyl-2-pentanone	0.100
Trichlorofluoromethane	0.100	Toluene	0.400
1,1-Dichloroethene	0.100	1,1,2-Trichloroethane	0.100
1,1,2-Trichloro-1,2,2-trifluroethane	0.100	Tetrachloroethane	0.200
Acetone	0.010	2-Hexanone	0.100
Carbon disulfide	0.100	Dibromochloromethane	0.100
Methyl Acetate	0.100	1,2-Dibromoethane	0.100
Methylene chloride	0.100	Chlorobenzene	0.500
Trans-1,2-Dichloroethene	0.100	Ethylbenzene	0.100
Cis-1,2-Dichloroethene	0.100	Meta-/para-Xylene	0.100
Methyl tert-Butyl Ether	0.100	Ortho-Xylene	0.300
1,1-Dichloroethane	0.200	Styrene	0.300
2-Butanone	0.01	Bromoform	0.100
Chloroform	0.200	Isopropylbenzene	0.100
1,1,1-Trichloroethane	0.100	1,1,2,2-Tetrachloroethane	0.300
Cyclohexane	0.100	1,3-Dichlorobenzene	0.600
Carbon tetrachloride	0.100	1,4-Dichlorobenzene	0.500
Benzene	0.500	1,2-Dichlorobenzene	0.400
1,2-Dichloroethane	0.100	1,2-Dibromo-3-chloropropane	0.050
Trichloroethene	0.200	1,2,4-Trichlorobenzene	0.200
Methylcyclohexane	0.100		

Table 11. Example EPA Method 8260D Analysis Sequence with QC and Instrument Calibration Frequency and Acceptance Criteria

Analysis #	Sample Name	QC and Instrument Calibration Acceptance Criteria	QC and Instrument Calibration Frequency
1	BFB	1. BFB see Section 17 Table 9	Required prior to instrument calibration only
2	Cal 1	1. Instrument Calibration must have %RSD ≤ 20%. If greater than 10% of analytes cannot meet the RSD or regression curve criteria (RSD < 20% or r^2 > 0.99), then instrument is out of control, and needs maintenance and recalibration. 2. Should meet 8260D recommended minimum RRF or be able to see standard at the reporting limit. 3. Must have relative retention time ± 017 RRT or ± 10 seconds	Calibration analyzed anytime CCV fails criteria and no obvious instrument problems can be found.
3	Cal 2		
4	Cal 3		
5	Cal 4		
6	Cal 5		
7	Cal 6		
8	Cal 7		
9	MB	Must be free from contamination that could prevent determination of target analytes at the RL. Must be < ½ the Project RL or < ½the LOQ,	Find problem and reanalyze all associated samples and QC.
10	SCV	1. Determination of target analytes 2. Concentration of target analytes must be within +/- 30% of true value. 3. IS Response 50 – 200% of Cal 3 or Cal midpoint 4. IS RT's ± 30 seconds	Analyzed immediately only after Cal curve
11		Jump to 13 after SCV.	SCV injection starts 12-hour clock
12	CCV	1. Percent Deviation of Target Analytes ± 20% 2. SS Percent Recovery--meet in-house limits. 3. Should meet 8260D recommended minimum RRF or evaluate to determine if the reporting limit can be achieved. 4. 3. IS Response 50 – 200% of o Cal midpoint 5. IS RT is within 30 seconds of calibration midpoint.	1. Analyzed initially with each batch of 20 samples within 12-hour work shift
13	MB	1. Same as Above	1. Same as above
14	MB Methanol	1. Same as Above	1. Same as Above
15	LCS/LCSD	1. Percent Recovery of Target analytes--meet in-house limits or data flagged. 2. SS Percent Recovery --meet in-house limits or data flagged. 3. IS Response 50 - +200% of CCV 4. RPD must meet in house limits	1. LCS is analyzed with each batch of 20 within a 12-hour work shift 2. LCSD analyzed only if no MS/MSD is in batch.
16	Sample 1	1. IS/SS see at the bottom of this table	
17	MS	1. Percent Recovery of Target Analytes--meet in-house limits or data flagged 2. SS Percent Recovery--meet in-house limits or data flagged 3. IS Response 50 - +200% of CCV	1. Analyzed with each batch of 20 within a 12-hour work shift
18	MSD	1 - 3 same as above %RSD (section 12) --meet in-house limits or data flagged	1. Analyzed with each batch of 20 within a 12-hour work shift
19	Samples 2-20	See statements below for IS and SS.	Reanalyze at a dilution until recoveries meet acceptance criteria.
Internal Standard (IS) and Surrogate Standard (SS) in all samples and QCs must meet the following acceptance criteria: 1 IS Response 50 - 200 % of the midpoint of the most recent calibration or the daily CCV. 2 SS must meet in-house limits or data flagged			

Table 12. Example Purge and Trap-GC/MS Settings for EPA Method 8260D

GC/MS Settings for EPA Method 8260				
OI 4100 Autosampler			OI 4760 Eclipse Purge and Trap	
	Water Only	Soil Only	Trap:	10
Needle Rinses	1	1	Sparge Mount	40
SAMA (ul)	2	0	Sample (purge):	35
SAMB (ul)	0	2	Sample (bake)	40
SAMC (ul)	0	0	Prepurge Time	Off
SAMD (ul)	0	0	Preheat Time	Off
Purge Time	11	11	Purge Time:	11
Desorb Time	0.7	0.7	Dry Purge	Off
P & T Rinses	1	1	Trap Temp	20
Rinse Water	Hot	Hot	Water Mgmt Temps:	
WSettle Time	5sec			
W Stir Time		0	Purge	120
Soil Preheat Stir	na	yes	Desorb	0
Soil PreheatTime	na	0.5min	Bake	240
Soil PH/PG Temp	na	40	Bake Time	4
Soil Stir	na	yes	Bake Temp	210
Lop Size	10	10	Desorb Time	0.7
GC ReadyPol.	Inverted	Inverted	Desorb Temp	190
			Desorb Preheat Temp	180
			Transfer Line Temp	140
			Valve Oven Temp	140
			Sparger	25ml
			Options	
			Drain on Start Up	On
			Purge on Bake	On
			Drain After Abort	On
			Sample Introduction	4100
			GC Ready	Normal
			SAMLV20 Pressure	8
GC Inlet			Column	
Front Inlet			Column 1	
Mode:		Split 75:1	Capillary Column	
Initial Temp:		185°C	Model Number:	RTX-Volatiles
Pressure:		7.91 psi	Max Temp:	280 °C
Total flow:		78.6 mL/min	Nominal Length:	30 m
Gas Type:		He	Nominal diameter:	0. mm
			Nominal film thickness:	1.0 µm
			Initial flow:	1.0 mL/min
			Mode:	Constant flow
			Inlet:	Front Inlet
			Outlet:	MSD
			Outlet pressure:	Vacuum

GC/MS Settings for EPA Method 8260				
GC Oven				
Oven:		Ramps:		
Initial Temp:	50°C	#Rate	Final Temp	Final Time
Initial Time:	0 min	1. 10°C/min	100°C	1.0 min
Maximum Temp:	280°C	2. 40°C/min	220°C	5.0 min
Equilibration Time:	0.5 min	Run Time:	14.00 min	
		Aux Temp:	230°C	
Mass Spectrometer				
Tune File:	bfb.u	scan rate:	3	
Acquisition Mode:	Scan			
MS Method:	8260 2019.M	MS Zone		
		MS Quad:		150°C
Scan Parameters		MS Source (detector):		250°C
Low Mass:	35	Transfer Line		230°C
High Mass:	270	Solvent delay:		0.5 min

Table 13. Example Quantitation Ions and Qualifiers

Analyte	RT (min)	Quant Ion	Q1	Q2	Q3
Fluorobenzene IS	3.99	96	77	/	/
Dichlorodifluoromethane	0.90	85	87	/	/
Chloromethane	0.98	50	52	/	/
Vinyl Chloride	1.02	62	64	/	/
Bromomethane	1.16	94	96	/	/
Chloroethane	1.22	64	66	/	/
Trichlorofluoromethane	1.28	101	103	/	/
1,1-Dichloroethene	1.51	96	61	63	/
Acetone	1.83	58	43	/	/
Iodomethane	1.59	142	127	/	/
Carbon Disulfide	1.54	76	78	/	/
Methylene Chloride	1.80	84	86	49	/
Methyl tert-Butyl Ether	1.84	73	41	57	43
trans-1,2-Dichloroethene	1.89	96	61	98	/
Di isopropyl ether	1.95	45	43	87	59
1,1-Dichloroethane	2.27	63	65	83	/
cis-1,2-Dichloroethene	2.67	96	61	98	/
2,2-Dichloropropane	2.76	77	97	/	/
2-Butanone	3.20	72	43	/	/
Ethyl tert Butyl Ether	3.25	59	87	57	41
Bromochloromethane	2.83	128	130	50	/
Chloroform	2.90	83	85	/	/
1,1,1-Trichloroethane	3.09	97	99	61	/
Tert Amyl methyl ether	3.13	73	55	43	87
1,1-Dichloropropene	3.22	75	110	77	/
Carbon Tetrachloride	3.02	117	119	121	/
1,2-Dichloroethane-*d*4	3.63	65	102	/	/
Benzene	3.48	78	/	/	/
1,2-Dichloroethane	3.72	62	98	/	/
Chlorobenzene-d_5 IS	7.27	117	/	/	/
Trichloroethene	4.21	95	97	130	132
1,2-Dichloropropane	4.84	63	65	112	/
Dibromomethane	4.72	93	95	174	/
Bromodichloromethane	4.93	83	85	127	/
cis-1,3-Dichloropropene	5.63	75	77	39	/
4-Methyl-2-pentanone	6.29	43	58	85	100
Toluene-d_8	5.82	98	/	/	/
trans-1,3-Dichloropropene	6.31	75	77	39	/

Analyte	RT (min)	Quant Ion	Q1	Q2	Q3
1,1,2-Trichloroethane	6.46	83	97	85	/
Tetrachloroethene	6.25	164	129	131	166
1,3-Dichloropropane	6.71	76	78	/	/
2-Hexanone	7.08	43	58	57	100
Dibromochloromethane	6.61	129	127	/	/
1,2-Dibromoethane	6.81	107	109	188	/
Chlorobenzene	7.29	112	77	114	/
1,1,1,2-Tetrachloroethane	7.32	131	133	119	/
Ethylbenzene	7.33	91	106	/	/
m,p-Xylene	7.43	106	91	/	/
o-Xylene	7.71	106	91	/	/
1,4-Dichlorobenzene-*d*4 IS	8.69	152	115	150	/
Styrene	7.75	104	78	103	/
Bromoform	7.75	173	175	254	/
Isopropylbenzene	7.92	105	120	77	/
BFB	8.07	95	174	176	/
n-Propylbenzene	8.16	91	120	/	/
Bromobenzene	8.13	156	77	158	/
1,1,2,2-Tetrachloroethane	8.21	83	131	85	/
1,2,3-Trichloropropane	8.27	75	77	/	/
2-Chlorotoluene	8.24	91	126	/	/
4-Chlorotoluene	8.34	91	126	/	/
1,3,5-Trimethylbenzene	8.28	105	120	/	/
tert-Butylbenzene	8.64	119	91	134	/
1,2,4-Trimethylbenzene	8.49	105	120	/	/
sec-Butylbenzene	8.54	105	134	/	/
1,3-Dichlorobenzene	8.65	146	111	148	/
4-Isopropyltoluene	8.62	119	134	91	/
1,4-Dichlorobenzene	8.69	146	111	148	/
1,2-Dichlorobenzene	8.9	146	111	148	/
n-Butylbenzene	8.83	91	92	134	/
1,2-Dibromo-3-Chloropropane	9.29	75	155	157	/
Hexachlorobutadiene	9.58	225	223	227	/
1,2,4-Trichlorobenzene	9.6	180	182	145	/
Naphthalene	9.74	128	/	/	/
1,2,3-Trichlorobenzene	9.83	180	182	145	/
Toluene	5.87	92	91	/	/

Table 14. 8260D Method Acceptance Criteria

Item	Measure	Action
Instrument Tune	Outside Acceptance Criteria	Re-tune.
	Repeated failure indicates a need for system maintenance.	Perform system maintenance and re-tune the instrument. No analyses should be performed until the system is tuned correctly. Tune with calibration only.
Internal Standard(s)--(IS)	50-200 % of the mid-point of the initial calibration standard or the daily CCV	If the nonconformance is on a calibration or QC sample, evaluate the system (repair) and reanalyze. Remake the standard if an error is suspected.
		If the nonconformance is on a field sample, reanalyze. If the reanalysis is within limits, report the results within limits. If the reanalysis is outside limits, dilute and reanalyze. Report the diluted results.
Response Factor(s)	≥ 0.01 for problem analytes such as ketones, and ≥ 0.05 for all other target analytes.	If a response factor is below acceptance criteria, then the system must be evaluated to make sure the analyte can be seen at the reporting limit. Recalibrate and reanalyze affected samples.
Initial Calibration (ICAL)	Average Response Factor > 20.0 % RSD	Evaluate points in the curve for use of linear or quadratic regression (r2 must be ≥ 0.99). Also evaluate upper and lower points for removal. Criteria still not met, recalibrate if compound is an analyte of interest.
ICAL Low Point Eval	Not within ± 50 % of True Value	Recalibrate if % deviation or drift is not met and compound is an analyte of interest.on recalculation with new curve
All ICAl points except low point	Not within +/- 30% of true value	Recalibrate if % deviation or drift is not met and compound is an analyte of interest.on recalculation with new curve
Second Source Calibration Verification (SCV)	Not within ± 30% of true value for deviation or drift	Recalibrate if % deviation or drift is not met and the compound is an analyte of interest.
Continuing Calibration Verification (CCV)	Not within ± 20 % of True Value	Evaluate the system for problems, correct method or standard, perform routine maintenance, etc. Reanalyze standard and if failure repeats, then analyze a new ICAL
Method Blank	Analyte concentration must be <1/2 the reporting limit for the project or <1/2 the LLOQ.	If the associated samples are non detect, no action is required. If the analyte(s) is detected in the sample, flag with a "b" or reanalyze. If the analyte level in the sample is 10 times or greater than the blank contamination, the results are not affected. Locate the source of the contamination.
Laboratory Control Spike (LCS)	% Recovery. Laboratory acceptance criteria are evaluated every six months. Acceptable values are stored in LIMS.	If the LCS % Recovery is high and the sample is non detect, no action is required. If the LCS is high and the sample has detects, renalyze the sample. If the LCS is low, the sample(s) should be reanalyzed.
Laboratory Control Spike Duplicate (LCSD)	Same criteria as the LCS with the addition of RPD. RPD acceptance criteria is evaluated every six months with values stored in LIMS.	% Recovery same as the LCS. If the RPD value is above the acceptance criteria in LIMS, then evaluate the system for possible problems. Reanalyze samples as necessary.
Matrix Spike(MS)	% Recovery. Laboratory acceptance criteria are evaluated every six months. Acceptable values are stored in LIMS.	If the % Recovery is outside laboratory acceptance criteria, evaluate the LCS. If the LCS is in control, then there is a possible matrix effect. The sample should be flagged appropriately.
Matrix Spike Duplicate (MSD)	Same criteria as the MS with the addition of RPD. Acceptance criteria are evaluated every six months with values stored in LIMS.	% Recovery same as the MS. If the RPD value is above the acceptance criteria in LIMS, then evaluate the system for possible problems. Reanalyze the MS/MSD samples if possible or flag the results.
Surrogate(s)	% Recovery. Laboratory acceptance criteria are evaluated every six months. Acceptable values are stored in LIMS.	If the % Recovery is outside laboratory acceptance criteria on a QC sample, evaluate the system. Surrogate recalibration may be necessary. Reanalyze the QC samples.
		If the % Recovery is on a client sample, reanalyze. If the % Recovery is within criteria, report the sample within limits. If the % Recovery outside criteria is confirmed, there is a matrix effect. Flag the results as estimated and report both results.

Figure 1. 50 µg/L 8260D Total Ion Chromatogram

```
File       :C:\MSDChem\1\DATA\073009\073009004.D
Operator   : AG
Acquired   : 30 Jul 2009  12:13 pm using AcqMethod 8260B2.M
Instrument :   APL01B
Sample Name: VSTD025
Misc Info  :
Vial Number: 4
```

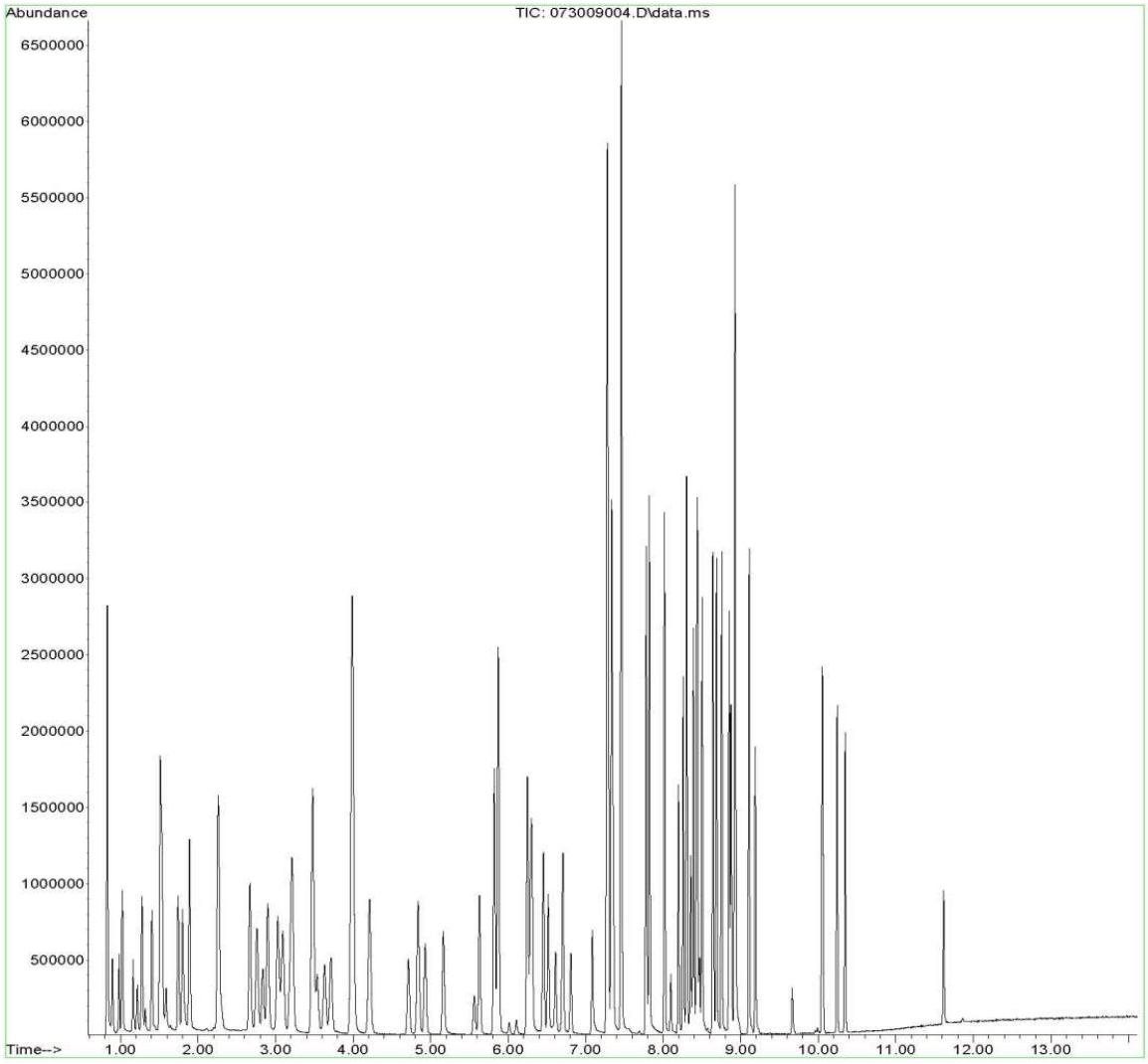

Figure 2. Example GC/MS Data Review Form

PHILIS Program

CSS

DATA REVIEW FORM – GC/MS

Instrument and Date: _____ **Sequence #:** _____

Analysis: *(Select One)* ☐ Semivolatiles ☐ Volatiles ☐ Other _____

	Yes	No	Peer Rvw	QA Rvw	Comments
Analyst Report					
PHILIS narrative is complete	☐	☐	☐	☐	
Reported data matches the raw data	☐	☐	☐	☐	
Reporting limits and qualifiers are correct	☐	☐	☐	☐	
Sample Receiving					
Samples received in acceptable condition and compliant with COC	☐	☐	☐	☐	
Samples properly preserved	☐	☐	☐	☐	
Sample receipt checklist filled out	☐	☐	☐	☐	
Instrument Tune and Calibration					
Instrument met tuning criteria, where required, and analyses were completed within the 12 hour clock	☐	☐	☐	☐	
ICAL average response factor % RSD is <20 or applied curve fit meets criteria	☐	☐	☐	☐	
The ICAL has an adequate number of points	☐	☐	☐	☐	
Response factors meet minimum criteria for ICAL and CCV	☐	☐	☐	☐	
ICAL low point is within 50% of known value and the mid-point is within 30% of the known value or SOP listed levels	☐	☐	☐	☐	
SCV is within 30 % of true values for deviation or drift	☐	☐	☐	☐	
CCV compounds meet acceptance criteria	☐	☐	☐	☐	
Method Blank					
Analytes detected at or above their reporting limits are flagged	☐	☐	☐	☐	
Samples					
Samples prepared and extracts analyzed within holding time limits	☐	☐	☐	☐	
Target compound report included and chromatograms provided	☐	☐	☐	☐	
Manual integration/Q-Deletion is initialed and dated by analyst and reviewer on ion profiles	☐	☐	☐	☐	
All target quantitation ion integrations and spectral identifications are included	☐	☐	☐	☐	
Calculations have been verified—see calculations sheet.	☐	☐	☐	☐	
Internal standard summary					
Is area between 50%-200% of the ICAL midpoint or daily CCV	☐	☐	☐	☐	
Retention times are within 0.5 minutes of the midpoint of the ICAL	☐	☐	☐	☐	
Surrogate recovery report					
Surrogate recovery meets acceptance criteria	☐	☐	☐	☐	
Sample results are properly flagged	☐	☐	☐	☐	
Preparation batch summary					
All samples are accounted for	☐	☐	☐	☐	
Results reflect sample mass/volume prepared	☐	☐	☐	☐	
Solid results are provided dry weight basis	☐	☐	☐	☐	
Matrix spike/matrix spike duplicate					
MS/MSD percent recoveries are within limits	☐	☐	☐	☐	
Relative percent differences are within limits	☐	☐	☐	☐	
Sample results been flagged appropriately	☐	☐	☐	☐	
Laboratory control spike/laboratory control spike duplicate					
LCS/LCSD recoveries are within limits	☐	☐	☐	☐	
Relative percent differences are within limits	☐	☐	☐	☐	
Have sample results been flagged appropriately	☐	☐	☐	☐	

Analyst review signature Date | Peer review signature Date | QA review signature Date

PHILIS2 Form ID#: QA-017 / Release Date: 09/18/2023

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