WSC – CAM – II B

Quality Control Requirements and Performance Standards for the ***Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

II. Gas Chromatography/Mass Spectrometry (GC/MS) Methods

B. Quality Control Requirements and Performance Standards for WSC-CAM-II B (Semivolatile Organic Compounds by GC/MS)

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ACRONYM LIST

Amu	**Atomic mass units**
CAM	**Compendium of Analytical Methods**
CASN	**Chemical Abstracts Service Number**
CCAL	**Continuing calibration**
%D	**Percent difference or percent drift**
DDT	**Dichlorodiphenyltrichloroethane**
DF	**Dilution factor**
DFTPP	**Decafluorotriphenylphosphine**
GC	**Gas chromatograph**
GC/MS	**Gas chromatography/mass spectrometry**
ICV	**Initial calibration verification**
IRAs	**Immediate Response Actions**
LCS	**Laboratory control sample**
LLE	**Liquid-liquid extraction**
LLOQ	**Lower limit of quantitation**
MassDEP	**Massachusetts Department of Environmental Protection**
MCP	**Massachusetts Contingency Plan**
MD	**Matrix duplicate**
mL	**Milliliters**
mL/min	**milliliters per minute**
MOHML	**Massachusetts Oil and Hazardous Materials List**
MS	**Matrix spike**
MSD	**Matrix spike duplicate**
MSE	**Microscale solvent extraction**
NA	**Not applicable**
PAHs	**Polycyclic aromatic hydrocarbons**
PFTBA	**Perfluorotributylamine**
PTFE	**Polytetrafluoroethylene**
QA	**Quality assurance**
QC	**Quality control**
r	**Correlation coefficient**
r^2	**Coefficient of determination**
RCs	**Reportable Concentrations**
RF	**Response factor**
RL	**Reporting limit**
RPD	**Relative percent difference**
RQs	**Reportable Quantities**
%RSD	**Percent relative standard deviation**
RT	**Retention time**
SIM	**Selective ion monitoring**
SPE	**Solid phase extraction**
SVOCs	**Semivolatile organic compounds**
TICs	**Tentatively identified compounds**
UCM	**Unresolved complex mixture**
µg/kg	**micrograms per kilogram**
µg/L	**micrograms per liter**
µL	**microliters**
VOCs	**Volatile organic compounds**

1.0 Quality Control Requirements and Performance Standards for WSC-CAM-II B

1.1 Overview of WSC-CAM-II B

WSC-CAM-II B, *Quality Control Requirements and Performance Standards for the Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective November 5, 2021, this revised CAM protocol, WSC-CAM-II B, replaces the previous version of the Semivolatile Organic GC/MS CAM document, WSC-CAM-II B (effective date, July 1, 2010). Refer to WSC-CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of November 5, 2021 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on July 22, 2021.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 8270E (or the most current version), analysis for semivolatile organic compounds (SVOCs) in aqueous and solid samples by GC/MS preceded by conventional sample preparation methods via SW-846 Methods, as described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table II B-1 together with the analytical procedures described in EPA SW-846 Method 8270E, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, constitute the WSC-CAM-II B protocol.

All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Since the analytical techniques for EPA SW-846 8270E, EPA SW-846 8270D, and EPA SW-846 8270C are substantially the same, use of any of these analytical methods (or a subsequent/more current version) meets the "Presumptive Certainty" requirement of WSC-CAM-II B.

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for SVOCs analyzed in support of MCP decision-making are presented in Appendix II B-1 of this document and Appendix VII-A of WSC-CAM-VII A *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*. Data reporting requirements are also provided in WSC-CAM-VII A.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits or Lower Limits of Quantitation for WSC-CAM-II B

The reporting limit (RL) or lower limit of quantitation (LLOQ) for an individual compound using WSC-CAM-II B is dependent on the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for the sample size, extraction concentration factor, percent solids, dilution factors, etc., as required. Except as noted below, the CAM RLs/LLOQs for WSC-CAM-II B target analytes using the full scan mode of operation are:

- 5-10 µg/L for aqueous samples (surface water, groundwater, and drinking water); and
- 330-670 µg/kg (wet weight) for soil/sediment samples (assuming 100% solids).

There may be exceptions to the above CAM RLs/LLOQs for some target analytes (that is, the CAM RL/LLOQ for some target analytes may not be readily achieved by a laboratory using WSC-CAM-II B). In general, aniline and substituted phenols will likely exhibit RLs/LLOQs 2-5 times higher than the CAM RLs/LLOQs listed above in both the solid and aqueous matrices.

The CAM RLs/LLOQs for the polycyclic aromatic hydrocarbons (PAHs), using the selective ion monitoring (SIM) mode of operation, are as follows:

- 0.1 µg/L for aqueous samples (surface water, groundwater, and drinking water); and
- 10 µg/kg (wet weight) for soil/sediment samples (assuming 100% solids).

For "Presumptive Certainty" purposes, if the CAM RLs/LLOQs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL/LLOQ exceedance in the laboratory narrative.

RLs/LLOQs lower than the above-referenced CAM RLs/LLOQs for WSC-CAM-II B target analytes may be required to satisfy project requirements. The RL/LLOQ (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require analytical modifications such as the use of SIM, an ion trap mass spectrometer, or other instrumentation of improved design to improve sensitivity. All such modifications must be described in the laboratory narrative. Regardless of the instrument that is used, RLs/LLOQs for the WSC-CAM-II B target analytes will be proportionately higher for samples that require dilution, when a reduced sample size is used, or for an increased final extract volume.

1.1.2 Initial Demonstration of Proficiency for WSC-CAM-II B

Each laboratory that uses the WSC-CAM-II B protocol is required to operate a formal quality assurance (QA) program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and the extraction/analysis of laboratory control samples (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or matrix duplicates (MDs) may also be used to evaluate accuracy and precision when such samples are analyzed either at the discretion of the laboratory or at the request of the data user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table II B-1 of this protocol and SW-846 Method 8000D. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000D (Section 9.3) and SW-846 method 8270E (Section 9.4). The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-II B must include the following information:

QC Element	Performance Criteria
DFTPP Tuning	WSC-CAM–II B, Table II B-1
Initial Calibration	WSC-CAM-II B, Table II B-1
Continuing Calibration	WSC-CAM-II B, Table II B-1
Method Blanks	WSC-CAM-II B, Table II B-1
Average Recovery	SW-846 Method 8000D, Section 9.3
% Relative Standard Deviation	SW-846 Method 8000D, Section 9.3
Surrogate Recovery	WSC-CAM-II B, Table II B-1
Internal Standards	WSC-CAM-II B, Table II B-1

NOTE: Because of the extensive analyte list and number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all non-conforming analytes. All non-conforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency documentation.

It is essential that laboratory-specific performance criteria for LCS, LCS duplicate and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000D, Section 9.6. Experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices; the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table II B-1 to assess analytical trends (i.e., systematic bias, etc.) and improve overall method performance by preempting potential non-conformances.

For the WSC-CAM-II B protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table II B-1. It should be noted that the performance standards listed in Table II B-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory.

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS instrumentation as a quantitative tool and skilled in the interpretation of chromatograms and mass spectra.

1.2 Summary of SW-846 Method 8270E

The samples are prepared for GC/MS analysis using the appropriate sample preparation and, if necessary, sample cleanup procedures (refer to Section 1.3).

Semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) equipped with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to facilitate separation of the analytes of interest, which are then detected by a mass spectrometer that is interfaced directly to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing sample electron impact mass spectra and retention times with electron impact mass spectra and retention times of standards obtained under identical analytical conditions. Quantitation is accomplished by using the response of a major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

In full scan operational mode, the mass spectrometer would typically acquire mass spectra from mass/charge 35 to 500 at a rate fast enough to acquire at least five (but preferably 10 or more) mass spectra across each chromatographic peak of interest. These parameters may vary depending on specific instrument capabilities. The SIM operational mode is used to increase sensitivity. In the SIM operational mode, the mass spectrometer repeatedly acquires mass spectra of a smaller number of pre-selected mass/charges rather than the typical mass/charge range utilized in the full scan mode. In the GC/MS-SIM acquisition mode, the mass/charges to be monitored are selected based on the mass spectra of compound(s) to be analyzed. The detector typically scans for a primary, secondary and tertiary set of mass/charges, unique to the compound of interest, in a particular retention time window. With more sophisticated instrumentation, mass/charges may be changed during the chromatographic run to accommodate multiple analytes, but with different retention times. GC/MS-SIM is a valuable tool for improving detection limits without compromising positive identification of analytes of concern. For some analytes, sensitivity may be increased by a factor of ten (10), as compared with a GC/MS system operated in the full scan mode.

1.3 Sample Extraction/Cleanup Methods for WSC-CAM-II B

Samples for analysis by SW-846 Method 8270E must be extracted or diluted using one of the following methods.

SW-846 Extraction Method	Matrix	Description
3510C	Aqueous	Separatory Funnel Liquid-Liquid Extraction (LLE)
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3535A	Aqueous	Solid-Phase Extraction (SPE)
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids[1]	Ultrasonic Extraction
3580A	Non-aqueous Phase Liquid	Waste Dilution
[1]Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed.		

In very limited applications, direct injection of an aqueous sample into the GC/MS system with a 10-µL syringe may be appropriate. The RL/LLOQ using this approach is very high (approximately 10,000 µg/L). Therefore, it is only permitted where concentrations in excess of 10,000 µg/L are expected.

Extracts may be cleaned up, as required, by any of the following methods prior to GC/MS analysis by SW-846 Method 8270E.

Analytes of Interest	SW-846 Cleanup Methods
Aniline & Aniline Derivatives	3620C
Phenols	3630C, 3640A, and 3650B
Nitrosamines	3610B, 3620C, and 3640A
Phthalate Esters	3610B, 3620C, and 3640A
Organochlorine Pesticides	3610B, 3620C, 3640A, and 3660B
Polychlorinated Biphenyls	3620C, 3630C, 3660B, and 3665A
Nitroaromatics and Cyclic Ketones	3620C and 3640A
Polynuclear Aromatic Hydrocarbons	3611B, 3630C, and 3640A
Haloethers	3620C and 3640A
Chlorinated Hydrocarbons	3620C and 3640A
Organophosphorus Pesticides	3620C and 3640A
Petroleum Wastes	3611B and 3650B
All Base-neutral and Acid Semivolatile Organics	3640A

1.4 Method Interferences

- Refer to SW-846 Methods 3500C (Section 4.0, in particular), 3600C, and 8000D for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

  - Contaminated solvents, reagents, or sample processing hardware,
  - Contaminated GC carrier gas, parts, column surfaces, or detector surfaces,
  - Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
  - Co-elution of target analytes.

  An in depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

- Refer to SW-846 Method 8270E for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination. If a method blank contains a contaminant, data for samples associated with that blank must **not** undergo "blank correction" (i.e., if an associated sample also contains the contaminant, subtraction of the blank amount from the sample amount is not permitted).

- Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of SVOCs. After the analysis of a sample containing high concentrations of SVOCs, one or more blanks should be analyzed to check for potential cross-contamination/carryover. Concentrations of SVOCs which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point

compounds may not appear until a later sample analysis. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

1.5 Quality Control Requirements for WSC-CAM-II B

1.5.1 General QC Requirements

Refer to SW-846 Method 8000D for general QC procedures for all chromatographic methods, which includes SW-846 Method 8270E. Instrument QC and method performance requirements for the GC/MS system may be found in SW-846 Method 8270E, Sections 9.0 and 11.0, respectively.

1.5.2 Specific QC Requirements and Performance Standards for WSC-CAM-II B

Specific QC requirements and performance standards for the WSC-CAM-II B protocol are presented in Table II B-1. Refer to WSC-CAM-VII A for field QC requirements. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty" for analytical data, parties must:

(a) Use the analytical method specified for the selected CAM protocol;
(b) Incorporate **all** required analytical QC elements specified for the selected CAM protocol;
(c) Implement, as necessary, required corrective actions and analytical response actions for **all** non-conforming analytical performance standards;
(d) Evaluate and narrate, as necessary, **all** identified CAM protocol non-compliances; and
(e) Comply with **all** the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of ten (10) years.

In achieving "Presumptive Certainty" status, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and

- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) and 40.1057(2)(k) for Permanent and Temporary Solution submittals, consistent with the guidance described in MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.6 Special Analytical Considerations for WSC-CAM-II B

The following bullets highlight potential issues that may be encountered with the analysis of SVOCs using this protocol.

- Because of the variable solubility, extraction efficiency and analytical sensitivity of the different classes of SVOCs that are potentially analyzable by SW-846 Method 8270E, the recovery ranges presented in Table II B-1 for LCSs, MSs, and surrogates should be considered general upper/lower acceptance limits when a single extraction procedure is utilized to prepare the extract for subsequent analysis.

- In some cases, the standard laboratory acceptance criteria for the various QC elements may have to be modified to accommodate more rigorous project-specific data quality objectives prescribed by the data-user. The laboratory may be required to modify routine pre-treatment, extraction, cleanup, sample introduction and/or analytical conditions to accommodate data quality objectives. Such cases include but are not limited to:

  - <u>Phenolic Compounds in Groundwater/Surface Water</u>.

    For health-based risk assessment decisions or compliance with cleanup standards, SW-846 Method 3510C (Separatory Funnel LLE) may not be suitable (or may not meet project-specific data quality objectives) for sample extraction because of known low recoveries (< 25%). For the phenolic compounds in groundwater or surface water, SW-846 Method 3520C (Continuous LLE) may be more suitable because of the improved recoveries (> 70%).

    In the presence of samples containing residual chlorine, the surrogate phenol-d6 may react to form chlorinated phenolic compounds. In order to minimize the degradation of this surrogate, proper preservation procedures must be utilized for samples containing residual chlorine, as described in Appendix II B-1.

  - <u>Loss of More Volatile Analytes During Extract Concentration Step</u>.

    More volatile analytes such as 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene, 1,4-dioxane, and naphthalene may be lost during the evaporation concentration step of the extraction procedure. As a result, many of the extraction methods listed in Section 1.3 may yield low recoveries for these analytes unless great care is exercised during the concentration steps. It may be appropriate to use additional surrogates which have similar physiochemical properties such as 1,2-dichlorobenzene-d4 or 1,4-dioxane-d8 to monitor for potential losses during the concentration steps.

  - <u>SVOCs in Soil/Sediment</u>.

    For health-based risk assessment decisions or compliance with cleanup standards, the recovery of these compounds from a soil matrix using SW-846 Method 3550C (Ultrasonic Extraction) may not be suitable because of insufficient recoveries (<40%) and low extraction efficiencies of this method. The more aggressive SW-846 Methods 3540C/3541 (Soxhlet Extraction), 3545A (Pressurized Fluid Extraction), or 3546 (Microwave Extraction) may be more suitable because of the improved recoveries (>70%).

  - <u>1,4-Dioxane in Groundwater/Surface Water.</u>

    If 1,4-dioxane is a contaminant of concern for the site, special analytical techniques, as listed below, must be utilized in order to evaluate compliance with MCP cleanup standards.

As described in Appendix II-B-4, 1,4-dioxane in groundwater/surface water may be analyzed in support of MCP response actions using GC/MS-SIM with isotopic dilution using 1,4-dioxane-d_8 as an extraction internal standard. Two separate extraction procedures (liquid-liquid and solid phase extraction) are described in the aforementioned appendix.

Heated (80±5°C) purge-and-trap with SIM analysis by SW-846 Method 8260D, as outlined in WSC-CAM-II A is an acceptable approach. However, if elevated concentrations of other volatile organic compounds (VOCs) are present in the sample, this approach may not be preferable due to the likely contamination/saturation of the trap during the analysis. The RL/LLOQ for 1,4-dioxane in aqueous samples is significantly lower (2 µg/L) using SW-846 method 8260D with heated (80±5 °C) purge-and-trap and GC/MS analysis in SIM mode, but still too high for analyses of samples in GW-1 areas or plumes that may migrate into GW-1 areas.

In the examples listed above, the data user must evaluate whether the analytical results based on the low recoveries associated with the more commonly used extraction procedure are suitable to verify compliance with project-specific data quality objectives. If not, a corrective action must be implemented to produce data of known accuracy and precision and suitable for the intended purpose. It should be noted that the recoveries attainable with the different extraction methods may vary between laboratories. Data users should discuss the use of specific extraction procedures with the laboratories prior to use to ensure that the data quality objectives can be achieved.

- A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation (%RSD) requirements specified in Table II B-1.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 9.3 of SW-846 8000D and Section 9.4 of SW-846 8270E.	No	NA	Refer to Section 9.3 of SW-846 8000D and Section 1.1.2 of this protocol.	NA
Annual LLOQ Verification	Sensitivity	(1) Must follow procedure in Section 9.7 of SW-846 8000D and Section 9.9.1 of SW-846 8270E.	No	NA	Recalibrate if needed or raise the LLOQ and repeat the verification.	NA
GC/MS Tunes with DFTPP	Inter-laboratory Consistency & Comparability	(1) Criteria listed in Table 3 of SW-846 8270E. (2) Prior to initial calibration. (3) DDT breakdown must be evaluated and must be ≤20%. (4) Pentachlorophenol and benzidine peak tailing must be evaluated. Peak tailing factor must be ≤2 for benzidine and pentachlorophenol. NOTE: Pentachlorophenol tailing must be evaluated when analyzing for acid SVOCs and benzidine tailing must be evaluated when analyzing for base-neutral SVOCs. These evaluations are not required if only analyzing for PAHs. (5) NOTE: Tune using DFTPP must be performed in full scan mode for SIM analyses. As an alternative to DFTPP for SIM analysis, an alternate reference compound, such as PFTBA, can be used. (6) Calibration standards and samples must be analyzed under the same tune	No	NA	Perform instrument/injection port maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing factor exceedances in laboratory narrative.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		conditions established prior to the initial calibration.				
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be ≤RL/LLOQ. (4) %RSD ≤20 (average RFs), r ≥0.995 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8270E for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples. (9) Target analyte peaks in the calibration standard at the RL/LLOQ should be visually inspected to ensure peak signal distinguishable from background and to verify acceptable qualitative analyte identification (e.g., RT and mass spectra). (10) If linear or non-linear regression used, verify the RL/LLOQ by recalculating	No	RF <0.01; affects nondetect results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if >10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. (3) If recalculated concentrations from the lowest calibration standard are outside of 50-150% recovery range, either: * The RL/LLOQ must be reported as an estimated value[2], or * The RL/LLOQ must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.995, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		concentrations in lowest calibration standard using the final calibration curve; recoveries must be 50-150%. (11) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards.				
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target analytes. (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes(**) which must exhibit percent recoveries between 40-160%.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, including "difficult" analytes(**), report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Percent difference or percent drift (%D) must be $\leq$20 for each target analyte except for "difficult" analytes(**) which must exhibit %Ds <60. (5) Minimum RFs as per Table 4 of SW-846 8270E.	No	RF <0.01; affects nondetect results for affected analyte in all samples analyzed under this continuing calibration.	(1) Recalibrate if >20% of all target analytes or >15% of analytes from a particular class (base-neutral or acid) exceed %D criteria. (2) If internal standard is outside of criteria, locate source of problem and re-analyze the continuing calibration. (3) If $\leq$20% of compounds exceed criteria,	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory narrative. Note in the laboratory narrative if the %D indicates a low or high bias.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(6) Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration standard. (7) In lieu of daily tune, monitor responses and chromatography in the continuing calibration for signs that the system is too reactive for analysis (e.g., ≥50% loss of reactive analytes, unusual tailing [tailing factor >2), loss of resolution).			recalibration is not required as long as %D <40 (or <60 for "difficult" analytes(**)). (4) If system is determined to be too reactive for analysis, perform maintenance and/or demonstrate that there is adequate sensitivity at the RL/LLOQ.	
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Per preparation batch of 20 or fewer samples. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <1/2 RL/LLOQ except for common laboratory contaminants (phthalates) which must be <5x the RL/LLOQ. (4) A method blank or instrument blank must be analyzed after the continuing calibrations.	Yes	NA	(1) If concentration of contaminant in sample is ≤10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blank is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Per preparation batch of 20 or fewer samples. (2) Concentration level near midpoint of curve. (3) Prepared using same standard source as used for initial calibration. (4) Must contain all target analytes. (5) Matrix-specific (e.g., water, soil). (6) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes(**) which must exhibit percent recoveries between 15-140% (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), re-extraction is not required if affected compounds were not detected in associated samples.	(1) if sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes(**), report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Per preparation batch of 20 or fewer samples. (2) Concentration level near midpoint of curve. (3) Prepared using same standard source as used for initial calibration. (4) Must contain all target analytes. (5) Matrix-specific (e.g., water, soil). (6) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes(**) which must exhibit percent recoveries between 15-140%. (7) RPDs must be ≤20 for waters and ≤30 for solids. (8) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), re-extraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes(**) or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Per preparation batch of 20 or fewer samples (at discretion of laboratory or at request of data user). (2) Matrix-specific (e.g., water, soil). (3) Concentration level near midpoint of curve.	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note non-conformances in laboratory narrative.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(4) Prepared using same standard source as used for initial calibration. (5) Must contain all target analytes. (6) Percent recoveries between 40 -140% for the base-neutral compounds and between 30-130% for the acid compounds. (7) RPDs ≤20 for waters and ≤30 for solids. (8) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).				
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral surrogates and 3 acid surrogates, at retention times across GC run. Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol NOTE: For SIM analyses, surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs and use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in solid matrices must be between 30-130% for individual surrogate compounds. Percent recoveries in water matrices must be between 30-130% for individual base-neutral surrogate compounds and 15-110% for individual acid surrogate compounds.	Yes	Recovery <10%; affects all nondetect SVOC results associated with the surrogate compound class (base-neutral or acid) in affected sample.	If two or more surrogates for any one class (base-neutral or acid) are outside of limits or if any one surrogate recovers at <10%: (1) Re-extract the sample if surrogate recoveries are low. (2) Re-extract the sample if surrogate recoveries are high and associated SVOCs were detected in the sample. Re-extraction is not required if one of the following exceptions applies: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required.	(1) Report recoveries outside of acceptance limits in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
					(b) Re-extraction is not required if obvious interference present (e.g., UCM). If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%), re-analyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, re-extraction and/or re-analysis is not required.	laboratory must report results of both extractions. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 6 at retention times across GC run. NOTE: For SIM analyses, the number of internal standards used will be dependent on the analytes of interest. Internal standards must elute in close proximity to the analytes of interest. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If one or more internal standards are outside of limits, re-analyze sample unless obvious interference present (e.g., UCM). NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), re-analyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If re-analysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Results must be reported with 2 or more "significant figures" if ≥RL/LLOQ. If reporting values below the RL/LLOQ, report with 1 or more "significant figures".[3]	NA	NA	NA	NA
Identification	NA	(1) Refer to SW-846 8270E, Section 11.6.	NA	NA	NA	NA

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(2) For SIM, use the midpoint of the initial calibration to establish acceptable ion ratios for each compound and to establish reference mass spectra.				
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific RL/LLOQ; optionally, values below the sample-specific RL/LLOQ can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Refer to Section 3.3, TICs by GC/MS, for guidance. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (5) Refer to Appendix II B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific RL/LLOQ. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix II B–3. (4) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (5) If samples are not properly preserved or are not received with an acceptable cooler

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270E) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						temperature, note the non-conformances in the laboratory narrative. (6) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

** Potentially "difficult" analytes include: dimethyl phthalate, 4-nitrophenol, phenol, 4- methylphenol, 2-methylphenol, 2,4-dinitrophenol, pentachlorophenol, and 4-chloroaniline

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2]If the RL/LLOQ is estimated due to unacceptable recovery of the lowest standard, the CAM RL/LLOQ has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

1.7 Analyte List for WSC-CAM-II B

The MCP analyte list for WSC-CAM-II B is presented in Table II B-2. The list is comprised of potential contaminants that are readily-analyzable by WSC-CAM-II B.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Reportable Concentrations (RCs) as described in 310 CMR 40.1600, The Massachusetts Oil and Hazardous Materials List (MOHML), in Subpart P of the MCP may be found at the following URL: http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/regulations/310-CMR-4000-massachusetts-contingency-plan

Most of the analytes listed in Table II B-2 have a promulgated MCP Method 1 groundwater/soil standard. The remaining analytes listed are designated “consensus contaminants” and do not have promulgated MCP Method 1 Standards as of the publication date of this revision.

1.7.1 Analyte List Reporting Requirements for WSC-CAM-II B

While it is not necessary to request and report all the WSC-CAM-II B analytes listed in Table II B-2 to obtain “Presumptive Certainty” status, it is necessary to document use and reporting of a reduced analyte list, for site characterization and data representativeness considerations. MassDEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830.

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note: a data user who avoids detection and quantitation of a contaminant that is present or likely present at a site above background levels by limiting an analyte list could be found in criminal violation of MGL c. 21E or any regulations or orders adopted or issued thereunder.

In cases where a reduced list of analytes is requested, laboratories must still employ the specified QC requirements and performance standards in WSC-CAM-II B to obtain “Presumptive Certainty” status.

Table II B-2: Analyte List for WSC-CAM-II B (SW-846 8270E)

Analyte	CASN	Analyte	CASN
Acenaphthene	83329	Dichlorobenzidine, 3,3'-	91941
Acenaphthylene	208968	Dichlorobenzene, 1,4-[1]	106467
Acetophenone	98862	Dichlorophenol, 2,4-	120832
Aniline	62533	Diethyl phthalate	84662
Anthracene	120127	Dimethyl phthalate	131113
Azobenzene	103333	Dimethylphenol, 2,4-	105679
Benzo(a)anthracene[1]	56553	Dinitrophenol, 2,4-	51285
Benzo(a)pyrene[1]	50328	Dinitrotoluene, 2,4-	121142
Benzo(b)fluoranthene[1]	205992	Dinitrotoluene, 2,6-	606202
Benzo(k)fluoranthene[1]	207089	Dioxane, 1,4-[1]	123911
Benzo(g,h,i)perylene	191242	bis (2-Ethylhexyl) phthalate[1]	117817
1,1'-Biphenyl	92524	Fluoranthene	206440
Bromophenyl phenyl ether , 4-	101553	Fluorene	86737
Butyl benzyl phthalate	85687	Hexachlorobenzene[1]	118741
Butyl phthalate, Di-n-	84742	Hexachlorobutadiene[1]	87683
Chloroaniline, 4-	106478	Hexachloroethane[1]	67721
bis (2-Chloroethoxy)methane	111911	Indeno (1,2,3-cd) pyrene[1]	193395
bis (2-Chloroethyl)ether	111444	Isophorone	78591
bis (2-Chloroisopropyl) ether	108601	Methylnaphthalene, 2-	91576
Chloronaphthalene, 2-	91587	Methylphenol, 2 -	95487
Chlorophenol, 2-	95578	Methylphenol, 3- [2]	108394
Chrysene[1]	218019	Methylphenol, 4- [2]	106445
Dibenz(a,h)anthracene[1]	53703	Naphthalene	91203
Dibenzofuran	132649	Nitrobenzene	98953
Dichlorobenzene, 1,2-	95501	Nitrophenol, 2-	88755
Dichlorobenzene, 1,3-	541731	Nitrophenol, 4-	100027
Di-n-octyl phthalate	117840	Pentachlorophenol[1]	87865

Table II B-2: Analyte List for WSC-CAM-II B (SW-846 8270E)

Analyte	CASN	Analyte	CASN
Phenol	108952	Phenanthrene	85018
Pyrene	129000	Trichlorophenol, 2,4,5-	95954
Trichlorobenzene, 1,2,4-	120821	Trichlorophenol, 2,4,6-	88062

[1]Standard RL/LLOQ for this compound may not be able to achieve regulatory compliance limit. A more effective extraction/sample introduction method or a more sensitive analytical procedure (e.g., SIM) may be required.

[2]3- and 4- Methylphenol (cresol) may co-elute.

CASN – Chemical Abstracts Service Numbers

NOTE: Other SVOCs may also be analyzed using the WSC-CAM-II B Protocol but are not considered part of the CAM target analyte list.

2.0 Data Usability Assessment

Specific guidance applicable to all Permanent and Temporary Solutions, including Permanent and Temporary Solutions on a portion of a disposal site, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively, of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-07-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of a Permanent or Temporary Solution submittal. The most current version of this document may be found at the following URL: http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-07-350, regardless of "Presumptive Certainty" status.

3.0 Reporting Requirements for WSC-CAM-II B

3.1 General Reporting Requirements for WSC-CAM-II B

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides limited recommendations for field QC, as well as the required content of the laboratory report, which includes:

- Laboratory identification information,
- Analytical results and supporting information,
- Sample- and batch-specific QC information,
- Laboratory Report Certification Statement,
- Copy of the Analytical Protocol Certification Form,
- Laboratory narrative contents, and
- Chain-of-custody form requirements.

3.2 Specific Reporting Requirements for WSC-CAM-II B

Specific QC requirements and performance standards for WSC-CAM-II B are presented in Table II B-1. Specific reporting requirements for WSC-CAM-II B are summarized below in Table II B-3 as "Required Analytical Deliverables (**YES**)". Requirements listed as "YES" must always be included as part of the laboratory deliverable for this method. It should be noted that data for those items listed as "NO" under "Required Analytical Deliverables" must be available for review during an audit and may also be requested for inclusion in the analytical deliverable on a client-specific basis.

Soil and sediment results must be reported on a dry-weight basis. Refer to ASTM Method D2216, Determination of Moisture Content of Soils and Sediments, for more detailed analytical and equipment specifications.

Table II B-3 Routine Reporting Requirements for WSC-CAM-II B (SW-846 8270E)

Parameter	Required Analytical Deliverable
GC/MS Tunes	***NO***
Initial Calibration	***NO***
Initial Calibration Verification	***NO***
Continuing Calibration (CCAL)	***NO***
Method Blank	***YES***
Laboratory Control Samples (LCSs)	***YES***
LCS Duplicates	***YES***
Matrix Spike (MS)	***YES (if requested by data user)***
Matrix Spike Duplicate (MSD)	***YES (if requested by data user)***
Matrix Duplicate (MD)	***YES (if requested by data user)***
Surrogates	***YES***
Internal Standards	***NO***
Tentatively Identified Compounds (TICs)	***YES (if requested by data user)***
Identification and Quantitation	***NO***
General Reporting Issues	***YES***

3.2.1 Sample Dilution

Under circumstances that sample dilution is required because either the concentration of one or more of the target analytes exceed the concentration of their respective highest calibration standard or any non-target peak exceeds the dynamic range of the detector (i.e., “off scale”), the RL/LLOQ for each SVOC must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL/LLOQ for the diluted sample, $RL/LLOQ_d$:

$$RL/LLOQ_d = DF \times \text{Lowest Calibration Standard for Target Analyte}$$

It should be understood that samples with elevated RLs/LLOQs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the $RL/LLOQ_d$ is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs/LLOQs are the unavoidable but acceptable consequence of sample dilution that enable quantification of target analytes which exceed the calibration range. All dilutions must be fully documented in the laboratory narrative.

NOTE: **Over dilution is an unacceptable laboratory practice.** The post-dilution concentration of the target analyte with the highest concentration must be at least 50% of its associated highest calibration standard. This will avoid unnecessarily high RLs/LLOQs for other target analytes which did not require dilution.

3.3 Tentatively Identified Compounds (TICs) by GC/MS

The evaluation of TICs in conjunction with GC/MS analyses (WSC-CAM-II A and WSC-CAM-II B) is a powerful and cost-effective analytical tool that can be particularly effective in assessing locations with suspect disposal practices, complex or uncertain site history, and/or sites that require detailed evaluation of critical exposure pathways. When GC/MS analytical methods are utilized in support of MCP decision-making, an analysis of TICs is:

Required when drinking water samples are analyzed (Refer to WSC-CAM-VII A for a definition of "drinking water"),
Should be considered in support of site characterization activities for releases at locations with complex and/or uncertain history,
Not usually expected at petroleum-only sites,
Not usually expected when the contaminants of concern have been previously identified, and/or
Not usually expected when used to determine the extent and magnitude of contamination associated with a "known" release of OHM.

It should be noted that TICs only need to be evaluated by the laboratory when specifically requested by the data user.

3.3.1 Reporting of TICs

If evaluated, all TICs that meet the chromatographic criteria presented in Section 1.0 of Appendix II B-3 must be reported by the laboratory either in the laboratory report or in the laboratory narrative. In turn, the data user must include a discussion regarding the disposition of all reported TICs as part of the MCP submittal. Depending on specific site circumstances (e.g., a potentially toxic contaminant is found in a private drinking water supply well, etc.), re-sampling/re-analysis with analyte-specific calibration and QC may be required to definitively assess the risk posed by the TIC to human health and the environment. Guidance for the evaluation of TICs for MCP decision-making is presented in Appendix II B-3 of this document.

Appendix II B-1

Sample Collection, Preservation, and Handling Procedures for Semivolatile Organic Compound Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for SVOCs analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*.

Matrix	Container[1]	Preservation[7]	Holding Time[3,6]
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to ≤ 6ºC but not frozen	7 days to extraction; 40 days from extraction to analysis[4]
Aqueous Samples, with Residual Chlorine[5]	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%)[5]. Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤ 6ºC but not frozen.	7 days to extraction; 40 days from extraction to analysis[4]
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap[2]	Cool to ≤ 6ºC[2]	14 days to extraction; 40 days from extraction to analysis[2,4]
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	Cool to ≤ 6ºC	14 days to extraction; 40 days from extraction to analysis[4]

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]Alternatively, soil/sediment samples for SVOC analyses may be held for up to one (1) year if frozen within 24 hours of collection at <-10°C. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Temperature must never be allowed to go below – 20°C to avoid damage to seals, etc. Preparation or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at 0-6°C until extraction.

[3]Holding time begins from time of sample collection or date thawed (see note #2 above).

[4]SVOC sample extracts must be stored at ≤6ºC, protected from light, and stored in sealed vials (e .g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa. See SW-846 Method 8270E, Section 8.2.

[5]Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine >5 mg/L, additional dechlorination agent may be required.

[6]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if the holding time is exceeded by >2x the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

[7]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6ºC are acceptable.

Appendix II B-2

Data Deliverable Requirements for Data Audits

If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a "full data package" including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated.

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-II B (SVOCs by GC/MS: SW-846 8270E)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences
Miscellaneous Logs	Dry weight logs Injection logs Soil/sediment sample weight logs Freezer logs Sample preparation/cleanup logs[1]
Initial Calibration Data	Summary of response factors for all standards in initial calibration; average response factors, %RSDs, correlation coefficients, and coefficients of determination for all target compounds Chromatograms for all standards used in initial calibration Quantitation reports for all standards used in initial calibration Concentrations of standards used must be clearly presented
Initial Calibration Verification Data	Summary of percent recoveries for all target compounds Chromatograms for all ICVs Quantitation reports for all ICVs
Continuing Calibration Data	Summary of %Ds and response factors Chromatograms for all continuing calibration standards Quantitation reports for all continuing calibration standards Concentrations of standards used must be clearly presented
Sample Results	Chromatograms for all sample analyses, re-analyses, and dilutions

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-II B (SVOCs by GC/MS: SW-846 8270E)	
	Quantitation reports for all sample analyses, re-analyses, and dilutions Mass spectra of reported positive results Percent solids results Summary of results, including RLs/LLOQs for each sample Date of analysis
Method Blank Results	Chromatograms for all method blanks Quantitation reports for all method blanks Summary of results, including RLs/LLOQs Mass spectra of positive results in method blanks Summary of how method blank was prepared in solid and aqueous matrices, as appropriate
LCS/LCS Duplicate Results	Chromatograms for all LCS and LCS Duplicates Quantitation reports for all LCS and LCS Duplicates Summary of results, including concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how LCS/LCS Duplicates were prepared in solid and aqueous matrices, as appropriate
MS/MSD Results (if performed)	Chromatograms for all MS/MSDs Quantitation reports for all MS/MSDs Summary of results, including unspiked sample concentrations, concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how MS/MSDs were prepared in solid and aqueous matrices, as appropriate
GC/MS Tune Data	DFTPP tune raw data: chromatogram, mass listing of DFTPP, and summary of tune results PFBTA tune results, if applicable
QC Summaries	Surrogate recoveries Internal standard performance
Other Information	Demonstration that ICV prepared from second source standard Summary of ions used for SIM analyses
Quantitation reports must exhibit area counts of target compounds, internal standards, and surrogates.	

DELIVERABLE REQUIREMENTS FOR DATA AUDITS **WSC-CAM-II B (SVOCs by GC/MS: SW-846 8270E)**
[1]Must clearly indicate sample weights or volumes, final extract volumes, extraction method used, extraction times where appropriate for the method, etc.

Appendix II B-3

Guidance for Evaluation of Tentatively Identified Compounds (TICs) for WSC-CAM-II B under the MCP

A logic diagram for the Evaluation of Tentatively Identified Compounds (TICs) for WSC-CAM-II B under the MCP is presented in Exhibit II B-1. This exhibit graphically presents a systematic approach to evaluate TICs based on chromatographic, mass spectral, and toxic spectral characteristics criteria.

1.0 Specific Criteria for the Evaluation of TICs

1.1 Chromatographic Criteria

- Initially include all of the non-target compounds that have a peak area count of ≥ 10% of the nearest internal standard.

1.2 Mass Spectral Criteria

- All spectra must be evaluated by a qualified mass spectrometrist.
- The spectral library match must be ≥ 80% for a tentative identification to be made.
- The major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum at similar relative intensities.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Major ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or for the presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks.
- Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different chromatographic retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs (as a mixture of two isomers).
- Spectra identified as “unknown” should be assigned to a general chemical class, if possible. Classification as a halogenated hydrocarbon, aldehyde/ketone, carboxylic acid, or cyano compound, etc. is acceptable. An explanation as to why more specific identification cannot be made (e.g., truncated spectra due to insufficient mass scanning range) must be provided in the laboratory narrative to support any “unknown” classification.
- TICs, which are identified as petroleum aliphatic hydrocarbons, do not have to be reported as TICs. However, there must be a statement in the laboratory narrative discussing the presence of these hydrocarbons in the sample(s).

After the above criteria are met, the top ten (10) compounds, chosen by comparing the area of the TIC to the area of the nearest internal standard, must be tentatively identified, quantitated, and reported.

1.3 Toxic Spectral Characteristics Criteria

Regardless of the peak area count in relation to the nearest internal standard, the laboratory must evaluate the spectra for any compound if the mass spectrum:

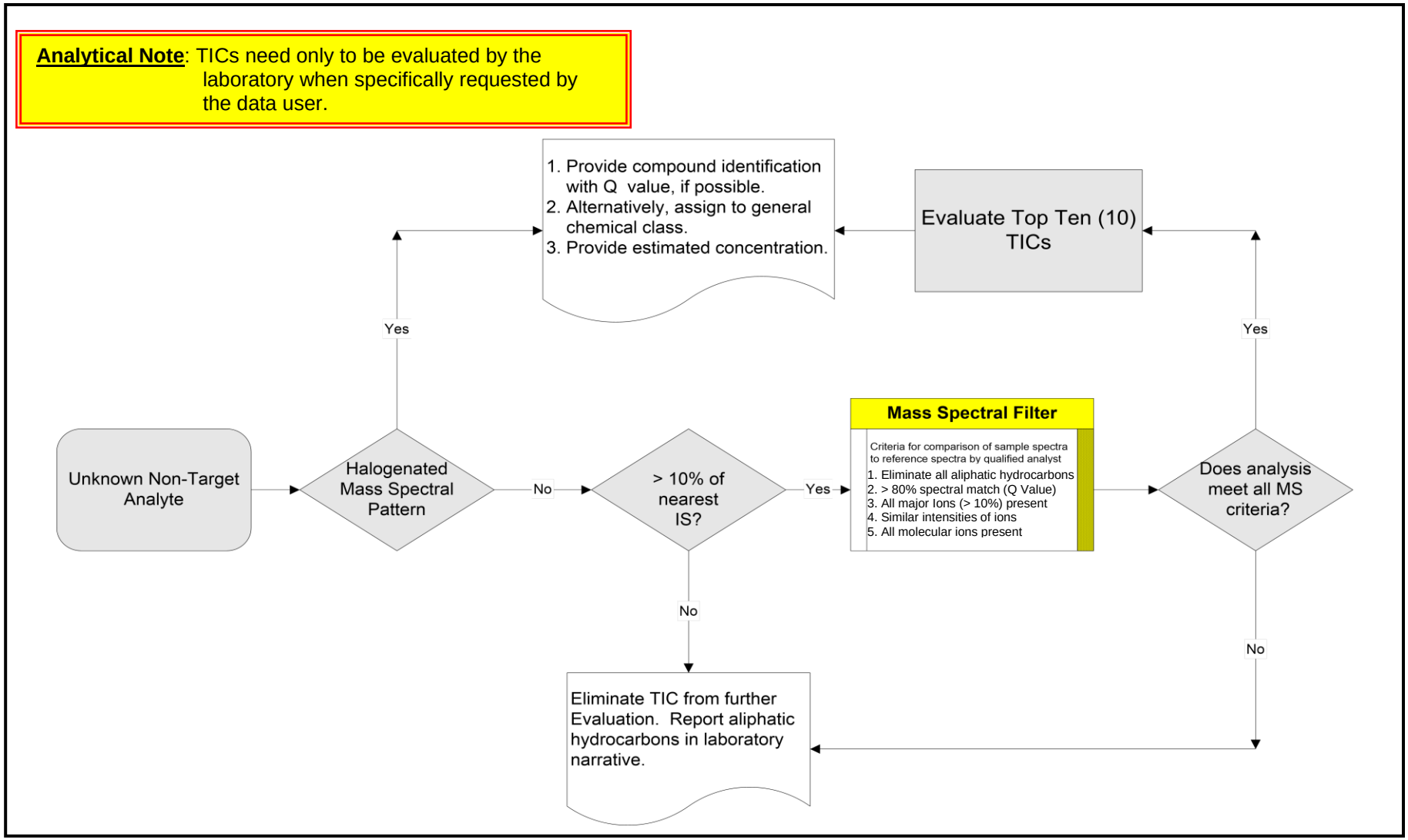
- Exhibits a characteristic chlorine or bromine spectral pattern.

2.0 Reporting Criteria

All TICs must be reported by the laboratory with the clear indication that the reported concentration is an estimated value unless analyte-specific calibration and QC were performed as discussed in Section 3.3.1. This reporting requirement may be fulfilled by discussion in the laboratory narrative, or by some other laboratory reporting convention to qualify the sample results. General laboratory reporting requirements are presented in WSC-CAM-VII A, Section 2.4.

If a data user determines that the presence of the TIC at the estimated concentration reported by the laboratory may appreciably increase the overall risk posed by the site or the utility/cost of the potential remedial measures under consideration, additional analytical work is recommended to verify the identification and/or concentration of the reported TIC either by re-analysis or re-sampling. This contingency will require additional coordination and communication between the laboratory and the data user.

Exhibit IIB-1



Appendix II B-4

Analytical Notes for 1,4-Dioxane Analysis for WSC-CAM-II B Under the MCP

A. SW-846 Method 3510C – Separatory Funnel Liquid-Liquid Extraction (LLE)

1. Methylene chloride extracts are analyzed using modified SW-846 Method 8270E GC/MS-SIM. 1,4-Dioxane-d_8 is added to the sample prior to extraction. This isotopically labeled compound is used as both an internal standard and a surrogate for the analyte of interest and serves to correct the variability associated with extraction of the target analyte using this extraction procedure. In turn, an additional internal standard, 1,4-Dichlorobenzene-d_4 is added post-extraction and is used to quantify 1,4-Dioxane-d_8 as the method surrogate.

2. This method is only applicable for the analysis of groundwater and surface water samples and has an achievable RL/LLOQ of 0.5 µg/L, or lower. Large volume injection techniques may be employed to achieve even lower RLs/LLOQs, if required.

3. Samples (1000 ml) are extracted using chromatographic grade methylene chloride LLE in a separatory funnel at a neutral pH in accordance with SW-846 method 3510C. The sample is extracted sequentially with three (3) volumes (60 mL) of methylene chloride. Subsequently, the combined extract volume is reduced to 1-5 ml prior to analysis.

4. A known concentration of 1,4-Dioxane-d_8 is added to the sample ***prior*** to LLE as an internal standard (extraction standard) for quantification of 1,4-Dioxane using GC/MS-SIM. A known concentration of 1,4-Dichlorobenzene-d_4 is added to the reduced-volume sample extract prior to analysis as an analytical internal standard and is used for the quantification of 1,4-Dioxane-d8.

   For GC/MS-SIM data acquisition, the following ions are suggested:

Compound	***Primary Ion***	***Secondary Ion***
1,4-Dioxane	88	58, 43
1,4-Dioxane-d_8	64	96
1,4-Dichlorobenzene-d_4	152	115

Unless otherwise specified in the following table, all Quality Control and Performance Standards specified in Table II-B-1 also apply to the analysis of groundwater and surface water samples using this modified LLE GC/MS-SIM method for "Presumptive Certainty" considerations.

Quality Control Criteria	**Performance Standard**
Initial Calibration	% RSD ≤ 20 or r ≥ 0.99
Continuing Calibration	%D ≤ 20
Area Counts of Analytical Internal Standard	Must be within –50 to +200% of the continuing calibration standard
Method Blank	1,4-Dioxane < RL/LLOQ
Extraction Internal Standard Recovery	15% - 110% Recovery
Laboratory Control Samples	40% - 140% Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140% Recovery; RPD ≤ 30

5. Identification criteria for 1,4-Dioxane include the following:

- Intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other;
- Retention time (RT) of the compound in the sample must be within 10 seconds of the RT of the compound in the standard; and
- Relative intensities of the characteristic ions must agree within ± 30% of the relative intensities of these ions in the reference spectrum.

B. SW-846 Method 3535 – Solid Phase Extraction (SPE)

Note: EPA Method 522 incorporates GC/MS-SIM and SPE for the analysis of 1,4-dioxane. For Presumptive Certainty purposes, this method is considered a modification of SW-846 Method 8270E, requiring a separate Initial Demonstration of Proficiency as described in the method.

1. Solid Phase Extraction tubes meeting the requirements of EPA Method 522 (Determination of 1,4-Dioxane in Drinking Water by Solid Phase Extraction [SPE] and Gas Chromatography/Mass Spectrometry [GC/MS] With Selected Ion Monitoring [SIM]) must be used for the extraction of 1,4-Dioxane. These commercially available tubes have a bed weight of 2 g of coconut charcoal and a tube volume of 6 mL.

2. Methylene chloride extracts are analyzed using modified SW-846 Method 8270E GC/MS-SIM. 1,4-Dioxane-d_8 is added to the sample prior to extraction. This isotopically labeled compound is used as both an internal standard and a surrogate for the analyte of interest and serves to correct the variability associated with extraction of the target analyte using this extraction procedure. In turn, an additional internal standard, Tetrahydrofuran-d_8 is added post-extraction and is used to quantify 1,4-Dioxane-d_8 as the method surrogate.

3. This method is only applicable for the analysis of groundwater and surface water samples and has an achievable RL/LLOQ of 1 µg/L, or lower. Large volume injection techniques may be employed to achieve even lower RLs/LLOQs, if required.

4. SPE tubes are installed on a standard SPE vacuum manifold. SPE tubes are cleaned and conditioned by sequential rinsing with three 5-mL aliquots of methylene chloride, followed by three 5-mL aliquots of methanol, followed by three 5-mL aliquots of reagent water. From the point of adding the last aliquot of methanol, the sorbent should not be allowed to go dry before the entire sample passes through the coconut charcoal tube. A thin layer of methanol or water should remain on top of the coconut charcoal tube.

5. A 500-mL water sample is added to the top of the "wetted" sorbent and filtered through the coconut charcoal tube at a rate of 5-10 mL/min using a vacuum. After the entire sample volume is processed through the coconut charcoal tube, a vacuum is pulled on the SPE tube for 5-10 minutes. The coconut charcoal tube is then eluted with 10 mL of methylene chloride. An aliquot of the extract is then transferred to a 2-mL autosampler vial. Because the extract contains some water, the extract must be dried with anhydrous sodium sulfate.

7. A known concentration of 1,4-Dioxane-d_8 is added to the sample ***prior*** to the SPE process as an internal standard (extraction standard) for quantification of 1,4-Dioxane using GC/MS-SIM. A known

concentration of Tetrahydrofuran-d_8 is added to the reduced-volume sample extract prior to analysis as an analytical internal standard and is used for the quantification of 1,4-Dioxane-d8.

For GC/MS-SIM data acquisition, the following ions are suggested:

Compound	***Primary Ion***	***Secondary Ion***
1,4-Dioxane	88	58, 43
1,4-Dioxane-d_8	64	96
Tetrahydrofuran-d_8	46	78, 80

Unless otherwise specified in the following table, all Quality Control and Performance Standards specified in Table II-B-1 also apply to the analysis of groundwater and surface water samples using this modified SPE GC/MS-SIM method for "Presumptive Certainty" considerations.

Quality Control Criteria	**Performance Standard**
Initial Calibration	% RSD ≤ 20 or r ≥ 0.99
Continuing Calibration	%D ≤ 20
Area Counts of Analytical Internal Standard	Must be within –50 to +200% of the continuing calibration standard
Method Blank	1,4-Dioxane < RL/LLOQ
Extraction Internal Standard Recovery	15% - 110% Recovery
Laboratory Control Samples	40% - 140% Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140% Recovery; RPD $\underline{\leq}$ 30

8. Identification criteria for 1,4-Dioxane include the following:

   - Intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other;
   - RT of the compound in the sample must be within 10 seconds of the RT of the compound in the standard; and
   - Relative intensities of the characteristic ions must agree within $\underline{+}$ 30% of the relative intensities of these ions in the reference spectrum.