



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

## **WSC – CAM - II B**

# **Quality Assurance and Quality Control Requirements for *SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)* for the Massachusetts Contingency Plan (MCP)**

**Document Status:** Final (1,4-Dioxane Notes)

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### **Document Chronology**

Revision	Status	Date
0	Public Comment Draft	31 August 01
1	Revised Public Comment Draft	5 October 01
2	Revised Interim Final	12 July 02
3	Final	1 May 2003
4	Final	13 August 2004
5	Final (1,4 Dioxane Notes)	20 December 2007



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

### **B. Quality Assurance/Quality Control (QA/QC) Requirements and Performance Standards for SW-846 Method 8270C (Semivolatile Organics by GC/MS)**

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## 1.0 QA/QC Requirements for SW-846 Method 8270C

### 1.1 Method Overview

SW-846 Method 8270C is used to determine the presence of semivolatile organic compounds (SVOCs) in a variety of matrices. With proper pretreatment and pre-concentration of samples (see Section 1.1.3 below), this method is applicable to nearly all types of environmental samples including various air sampling media, groundwater and surface water, soils, and sediments. All references to SW-846 methods in this document refer to the United States Environmental Protection Agency's most recently promulgated version.

SW-846 Method 8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic, fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, aromatic nitro compounds, and phenols.

In most cases, SW-846 Method 8270C is not appropriate for the quantitation of multi-component analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for these analytes. When these analytes have been identified by another technique, SW-846 Method 8270C is appropriate for **confirmation** of the presence of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of SW-846 Methods 8081A and 8082 for guidance on calibration and quantitation of multi-component analytes such as the Aroclors, Toxaphene, and Chlordane.

A number of specific analytes and classes of compounds, including benzidine, pyridine, toluene diisocyanate, phenolic compounds, and some nitrosamines may require special care and treatment when being determined by this method. Refer to SW-846 Method 8270C, Section 1.4 for details.

#### 1.1.1 Reporting Limits for SW-846 Method 8270C

The reporting limit (RL) using SW-846 Method 8270C for an individual compound is dependent on the concentration of the lowest analytical standard in the initial calibration, choice of sample preparation/introduction method, and/or percent (%) solids of the sample. Using standard quadrupole instrumentation, the reporting limit should be approximately 330 µg/kg (wet weight) for soil/sediment samples, 1 to 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Somewhat lower RLs may be achieved using selective ion monitoring (SIM), an ion trap mass spectrometer, or other instrumentation of improved design. Regardless of the instrument that is used, reporting limits for SW-846 Method 8270C will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

Sample preservation, container and analytical holding time specifications for surface water, groundwater, 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, WSC-CAM-



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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)".

1.1.2 General Quality Control Requirements of SW-846 Method 8270C

Each laboratory that uses SW-846 Method 8270C is required to operate a formal quality assurance 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 analysis of laboratory control spikes (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or Matrix duplicates may also be used to evaluate precision when such samples are analyzed either at 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 Section 1.4 and Table II B-1 of this document. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 method 8000B (Section 8.4) and SW-846 method 8270C (Section 8.3). The data associated with the Initial Demonstration of Proficiency should 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 SW-846 Method 8270C must include the following:

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 8000, Section 8.4
% Relative Standard Deviation	SW-846 Method 8000, Section 8.4
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 identify 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 data provided.



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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 8000B, Section 8.7. When 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 quality control 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 SW-846 Method 8270C, 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. Laboratories are encouraged to continually strive to minimize variability and improve the accuracy and precision of their analytical results. In some cases, the standard laboratory acceptance criteria for the various QC elements may require modification to accommodate more rigorous project-specific data quality objectives prescribed by the data user. The laboratory may be required to modify sample extraction or blow-down volumes and/or analytical conditions to accommodate project-specific data quality objectives.

This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers (GC/MS) as a quantitative tool and skilled in the interpretation of chromatograms and mass spectra.

### 1.1.3 Sample Extraction/Cleanup Methods for SW-846 Method 8270C

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

SW-846 Method	Matrix	Description
3542	Air Samples	Extraction of Analytes Collected Using a Modified Method 5 Sampling Train
3510C	Aqueous	Separatory Funnel liquid-Liquid Extraction
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction (PFE)
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids <sup>1</sup>	Ultrasonic Extraction
3580A	NAPL	Solvent 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**



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In very limited applications, direct injection of an aqueous sample into the GC/MS system with a 10- $\mu$ L syringe may be appropriate. The detection limit is very high (approximately 10,000  $\mu$ g/L). Therefore, it is only permitted where concentrations in excess of 10,000  $\mu$ g/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 8270C.

<b>Analytes of Interest</b>	<b>Cleanup Methods</b>
Aniline & Aniline Derivatives	SW-846 Method 3620
Phenols	SW-846 Methods 3630, 3640, and 8041 (derivatization)
Nitrosamines	SW-846 Methods 3610, 3620, and 3640
Phthalate Esters	SW-846 Methods 3610, 3620, and 3640
Organochlorine Pesticides & PCBs	SW-846 Methods 3610, 3620, 3630, and 3660
Polychlorinated Biphenyls (PCBs)	SW-846 Methods 3610, 3620, 3630, 3660, and 3665
Nitroaromatics and Cyclic Ketones	SW-846 Methods 3620 and 3640
Polynuclear Aromatic Hydrocarbons	SW-846 Methods 3611, 3630, and 3640
Haloethers	SW-846 Methods 3620 and 3640
Chlorinated Hydrocarbons	SW-846 Methods 3620 and 3640
Organophosphorus Pesticides	SW-846 Method 3620
Petroleum Wastes	SW-846 Methods 3611 and 3650
All Base, Neutral, and Acid Priority Pollutants	SW-846 Method 3640

## 1.2 Summary of Method

### 1.2.1 GC/MS System Operating in the Full Scan Mode

The samples are prepared for GC/MS analysis using the appropriate sample preparation and, if necessary, sample cleanup procedures (refer to Section 1.1.3 above).

The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph equipped with a narrow-bore fused-silica capillary column. The GC oven is temperature-programmed to facilitate separation of the analytes of interest, which are then detected by a mass spectrometer that is interfaced to the gas chromatograph. In a full scan operational mode,



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the mass spectrometer would typically scan a mass range of 35 to 500 atomic mass units (amu) at a frequency of 1 mass range scan/second. These parameters may vary depending on specific instrument capabilities.

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

### 1.2.2 GC/MS System Operating in the Selective Ion Monitoring (SIM) Mode

A GC/MS system is generally operated in the SIM mode to increase sensitivity. In the SIM mode, the mass spectrometer repeatedly scans a smaller number of pre-selected masses rather than the typical mass range (35 to 500 amu) utilized in the full scan mode. In the GC/MS SIM acquisition mode, the masses 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 masses, unique to the compound of interest, in a particular retention time window. With more sophisticated instrumentation, masses may be changed during the chromatographic run to accommodate multiple analytes, but with different retention times. GC/MS SIM is an invaluable 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.

Sample preparation, chromatographic conditions, analyte identification, and analyte quantification are the same whether the GC/MS system is operated in the full scan or SIM mode.

## 1.3 Method Interferences

### 1.3.1 Chemical Contaminants

Major contaminant sources for SW-846 Method 8270C include, but are not limited to, contaminated solvents and inadvertent contact of extraction fluids with rubber and/or plastic materials. The use of non-polytetrafluoroethylene (PTFE) thread sealants or plastic tubing should be avoided. It should be noted that interfering contaminants may also be concentrated during sample preparation and cleanup. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should review sample pretreatment and concentration procedures to determine the cause of the contamination before re-extraction occurs. **Subtracting blank values from sample results is not permitted.** If the laboratory determines that the concentration reported in the blank is so high that false positive results are likely in the associated samples, then the laboratory should fully explain this situation in the case narrative.

### 1.3.2 Cross-Contamination/Carryover

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of semivolatile organic compounds. After the analysis of a sample containing high concentrations of semivolatile organic compounds, one or more blanks should be



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analyzed to check for potential cross-contamination/carryover. The concentration of SVOCs which can cause cross-contamination/carryover must be determined by the laboratory and will be dependent upon the concentration and level of saturation of the particular analyte. 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 run. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

#### 1.4 Quality Control Requirements for SW-846 Method 8270C

##### 1.4.1 General Quality Control Requirements for Determinative Chromatographic Methods

Refer to SW-846 Method 8000B for general quality control procedures for all chromatographic methods, including SW-846 Method 8270C. These requirements ensure that each laboratory maintain a formal quality assurance program and records to document the quality of all chromatographic data.

Quality Control procedures necessary to evaluate the GC system operation may be found in SW-846 Method 8000B, Sec. 7.0, and include evaluation of calibrations and chromatographic performance of sample analyses. Instrument quality control and method performance requirements for the GC/MS system may be found in SW-846 Method 8270C, Sections 8.0 and 9.0, respectively.

##### 1.4.2 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8270C

Specific QA/QC requirements and performance standards for SW-846 Method 8270C when the GC/MS system is operated either in the full scan mode or the SIM mode are presented in Table II B-1. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying analytical and reporting requirements will provide an LSP with "Presumptive Certainty" regarding the usability of analytical data to support MCP decisions. 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", parties must:

- (a) Comply with the procedures described and referenced in WSC-CAM-II B;
- (b) Comply with the applicable QC analytical requirements prescribed in Table II B-1 for this test procedure;
- (c) Evaluate, and narrate, as necessary, compliance with performance standards prescribed in Table II B-1 for this test method; and



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(d) Adopt the reporting formats and elements specified in the CAM

*In achieving the status of "Presumptive Certainty", 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;
- ✓ May be used in a data usability assessment, and, if in compliance with all MCP Analytical Method standards, laboratory QC requirements, and field QC recommended limits and action levels, the data set will be considered usable data to support site characterization decisions made pursuant to the MCP; and
- ✓ May be used to support a data representativeness assessment.

#### 1.4.3 Special Analytical Considerations for SW-846 Method 8270C

Because of the variable solubility, extraction efficiency and analytical sensitivity of the different classes of semivolatile compounds that are potentially analyzable by SW-846 Method 8270C, the recovery ranges presented in Table II B-1 for laboratory control samples, matrix spikes, and surrogates should be considered general upper/lower acceptance limits when a single extraction procedure is utilized to prepare the extract for subsequent analysis. It is essential that laboratory-specific performance criteria for LCS and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. When 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 quality control 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.

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:

- Phenolic compounds are contaminants of concern in groundwater.

For health-based risk assessment decisions or compliance with cleanup, SW-846 Method 3510 (Separatory Funnel Extraction) 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, SW-846 Method 3520 (Continuous Liquid/Liquid Extraction) may be more suitable because of the improved recoveries (> 70%).



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➤ Semivolatile Organics in soil are contaminants of concern.

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

➤ 1,4-Dioxane in Groundwater

As described in Appendix II-B-3, 1,4-Dioxane in groundwater may be analyzed in support of MCP response actions using GC/MS-SIM with isotopic dilution using 1,4-Dioxane-d8 as an internal standard (IS). Two separate extraction procedures (liquid-liquid and solid phase extraction) are described in the aforementioned appendix. The analysis of soil and sediment samples for 1,4-Dioxane in support of MCP response actions is described in MCP Analytical Method II A, SW-846 Method 8260B, Volatile Organics by GC/MS (Final, August 2004)

In these examples, the LSP 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. LSPs should discuss the use of specific extraction procedures with the laboratories prior to use to ensure that the data quality objectives can be achieved.



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Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with DFTPP	Inter-laboratory consistency and comparability	(1) Criteria for DFTPP listed in Table 3 of SW-846 8270C (the same criteria must be used for all analyses) (2) Every 12 hours (3) DDT breakdown should be evaluated and should be <20%. (4) Pentachlorophenol and benzidine peak tailing should be evaluated. Peak tailing factor must be <3 for benzidine and <5 for pentachlorophenol. NOTE: Tune must be performed in full scan mode for SIM analyses.	No	Perform instrument/injection port maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing exceedances in the case narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be $\leq$ reporting limit (3) Full scan: %RSD should be $\leq 15$ or "r" should be $\geq 0.99$ for all compounds except CCCs which must be $\leq 30$ % RSD or "r" $\geq 0.99$ SIM: %RSD should be $\leq 20$ or "r" should be $\geq 0.99$ for all compounds (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin. (6) 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	No	Recalibrate as required by method (1) if any of CCC %RSDs >30 or any of CCC "r" <0.99 or (2) if >20% of remaining analytes have %RSDs >30 or "r" <0.99.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) Full scan: Percent difference or percent drift must be $\leq 20$ for CCCs and should be $\leq 30$ for other compounds SIM: Percent difference or percent drift should be $\leq 30$ for all compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20 or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in case narrative.



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Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent (2) Matrix-specific (e.g., water, soil) (3) Target analytes must be $\leq$ RL except for common laboratory contaminants (such as phthalates) which must be $<5x$ RL	Yes	Locate source of contamination; correct problem; re-extract associated samples if uncommon contaminants are present in the method blank.	(1) Report non-conformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the case narrative. (3) If re-extraction is performed within holding time, 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.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level should be between low and mid-level standard (4) Must contain all target analytes (5) Matrix-specific (e.g., soil, water) (6) Percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	Recalculate the percent recoveries; Re-extract associated samples if $>20\%$ of all analytes fall outside the acceptance criteria or if $>15\%$ of analytes from a particular class (base-neutral or acid) fall outside the acceptance criteria.	(1) Report non-conformances in case narrative. (2) Individual laboratories should identify and document "difficult" (***) analytes for which laboratory-determined recovery ranges routinely exceed the $100 \pm 30\%$ criterion. Exceedances for these "difficult" analytes should be qualified in case narrative. Analytical data to support the "difficult" analyte classification are to be available for review during an audit. (3) If re-extraction is performed within holding time, 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.
LCS Duplicate	Laboratory Method Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory-determined percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (6) Matrix-specific (e.g., soil, water, etc.) (7) Laboratory-determined Relative Percent Difference (RPD) must be $\leq 20$ for waters and $\leq 30$ for solids except for "difficult" (***) analytes which must be $\leq 50$ .	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined RPDs routinely exceed the $\leq 25$ criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances



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Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level should be between low and mid-level standard (5) Must contain all target analytes. (6) Percent recoveries should be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds, or develop laboratory in-house limits. (7) RPDs should be ≤20 for waters and ≤30 for solids	Yes  Only when requested by the data-user,	Check LCS; if recoveries acceptable in LCS, evaluate alternate cleanup techniques for samples associated with MS/MSD and/or narrate non-conformance.	Note exceedances in case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral and 3 acid, at retention times across GC run Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol <i>SIM Note:</i> Surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs, use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in soil must be between 30-130 for all surrogates. Percent recoveries in water must be between 30-130 for base-neutral surrogates and between 15-110 for acid surrogates. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	If two or more surrogates for any one fraction (base-neutral or acid) are outside limits or if any one surrogate recovers at <10%, reextract the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.	(1) Note exceedances in case narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory should 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 holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) <i>Full scan</i> : Minimum of 6 at retention times across GC run. <i>SIM</i> : 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 (Section 5.4.2 of 8270C) (3) Retention times of internal standards must be within $\pm 30$ seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, re-analyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in case narrative. (2) If re-analysis yields similar internal standard non-conformances, the laboratory should report both results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte (3) The internal standard used for quantitation shall be the one nearest the retention time of the subject analyte.	NA	NA	If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the case narrative with a list of the affected analytes.



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	NA	<p>(1) The laboratory must only report values <math>\geq</math> the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner.</p> <p>(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</p> <p>(3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance</p>			<p>(1) Qualification of the data is required if reporting values below the sample-specific reporting limit.</p> <p>(2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit.</p> <p>(3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II B-3.</p> <p>(4) The performance of dilutions must be documented in the case narrative.</p>

GC/MS = Gas Chromatography/Mass Spectrometry  
DFTPP = Decafluorotriphenylphosphine  
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates  
%RSD = Percent Relative Standard Deviation  
UCM = Unresolved Complex Mixture

"r" = Correlation Coefficient  
CCCs = Calibration Check Compounds  
RPDs = Relative Percent Differences  
TIC = Tentatively Identified Compound  
NA = Not Applicable

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



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

### 1.5 Analyte List for SW-846 Method 8270C

The MCP analyte list for SW-846 Method 8270C, presented in Table II B-2, is intended to be protective of human health and the environment. The list is comprised of potential contaminants that are readily analyzable by SW-846 Method 8270C and have compound-specific MCP Method 1 Groundwater/Soil Standards as described in 310 CMR 40.0974 and 40.0980, respectively. The remaining semi-volatile compounds that comprise the SW-846 Method 8270C Analyte List are designated "consensus contaminants". These semi-volatile compounds do not have a promulgated MCP Method 1 Standards but do have MCP Reportable Concentrations (RCs) as described in 310 CMR 40.0360 and 40.1600 and published EPA Integrated Risk Information System (IRIS) toxicity values. Using available toxicity data for these "consensus contaminants", the Department has derived compound-specific MCP Method 2 Groundwater/Soil Standards as described in 310 CMR 40.0983 and 40.0984, respectively. An updated list of the Department-derived MCP Method 2 Standards may be found at the following URL:

<http://www.mass.gov/dep/bwsc/files/standard/method2/method2.htm>

The MCP Method 1 Groundwater/Soil Standards used to characterize the risk of harm posed by oil or hazardous materials at a disposal site are described in 310 CMR 40.0974(2), Table 1. This list of groundwater/soil standards, developed by the Department, takes into account a defined set of conservative potential exposure pathways likely to be encountered at most disposal sites. Method 1 Standards have been developed by the Department for over one hundred organic and inorganic contaminants that are commonly encountered at disposal sites. The MCP Method 1 Groundwater/Soil Standards list is periodically reviewed and updated by the Department. When compounds are added to the MCP Method 1 Groundwater/Soil Standards list that are suitable for analysis by SW-846 Method 8270C, the analyte list for this method will be updated accordingly.

MCP Method 2 Groundwater/Soil Standards are developed by the Department (or others) for contaminants of concern for which MCP Method 1 Standards have not been promulgated. The use of Department-developed MCP Method 2 Standards is discretionary. Alternatively, site-specific MCP Method 2 Standards may be developed or a Method 3 risk characterization, as described in 310 CMR 40.0990, may be conducted to evaluate or characterize the risk of harm posed by oil or hazardous materials at a disposal site.

Additional analytes with promulgated MCP Method 1 Groundwater/Soil Standards, even though potentially analyzable by SW-846 Method 8270C, have not been included on this list. Classes of these potentially analyzable compounds excluded from the list include:

- Chlorinated Pesticides,
- Polychlorinated Biphenyls (PCBs),
- Non-Chlorinated Pesticides, and
- Other Miscellaneous Semivolatile Organics



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

These potentially-analyzable SW-846 Method 8270C analytes have been excluded from the list in some cases because of elevated detection limits, their rare occurrence at MCP disposal sites, the analytes are more easily analyzed by other methods, and/or unavailability of a published EPA IRIS toxicity value.

#### 1.5.1 Analysis of N-Nitrosodimethylamine by SW-846 Method 8270C

Although MCP Method 1 Groundwater/Soil Standards have been promulgated by the Department for N-Nitrosodimethylamine (CASN: 62-75-9), this compound is not included on the analyte list for SW-846 Method 8270C. N-Nitrosodimethylamine is difficult to separate from the solvent peak under specified method chromatographic conditions. See Section 1.4.4 of SW-846 Method 8270C. If this compound is a contaminant of concern at a site, either modified chromatographic conditions or an alternative analytical method (SW-846 Method 8070A, Nitrosamines by Gas Chromatography) may be employed to satisfy due diligence requirements and evaluate compliance with regulatory limits for this compound.

#### 1.6 Additional Reporting Requirements for SW-846 method 8270C

While it is not necessary to request and report all the SW-846 Method 8270C analytes listed in Table II B-2 to obtain "Presumptive Certainty" status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP 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:

- ✓ Uncharacterized 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.



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*Note that a desire to avoid detection and quantitation of a contaminant that is present or likely present at a site above background levels is not a valid reason to limit an analyte list, and that such an action could constitute a criminal violation of MGL c. 21E.*

In cases where a truncated list of method analytes is selected, laboratories must still employ the method-specific quality control requirements and performance standards associated with the requested analytes list to obtain "Presumptive Certainty" status.

The Reporting Limit (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, RfDs, benchmark values, background, etc.) with the exceptions footnoted in Table II A-2. Meeting "MCP program" reporting limits may require analytical modifications, such as increased sampling weight or volume or the use of selective ion monitoring, to increase sensitivity. All such modifications must be described in the Environmental Laboratory case narrative.



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Analyte	CASN	MCP CLEANUP STANDARDS	
		GW-1 (GW-3)	S-1/GW-1 (S-1/GW-3)
		µg/L (ppb)	µg/g (ppm)
<b>Table II B-2 Analyte List for SW-846 Method 8270C (Page 1 of 3)</b>			
Acenaphthene	83-32-9	20	20
Acenaphthylene	208-96-8	300	100
Acetophenone	98-86-2	X <sup>1</sup>	X <sup>1</sup>
Aniline	62-53-3	X <sup>1</sup>	X <sup>1</sup>
Anthracene	120-12-7	2000	1000
Azobenzene	103 -33-3	X <sup>1</sup>	X <sup>1</sup>
Benzo(a)anthracene	56-55-3	1 <sup>2</sup>	0.7
Benzo(a)pyrene	50-32-8	0.2 <sup>2</sup>	0.7
Benzo(b)fluoranthene	205-99-2	1 <sup>2</sup>	0.7
Benzo(k)fluoranthene	207-08-9	1 <sup>2</sup>	7
Benzo(g,h,i)perylene	191-24-2	300	1000
Bromophenyl phenyl ether , 4-	101-55-3	X <sup>1</sup>	X <sup>1</sup>
Butyl benzyl phthalate	85-68-7	X <sup>1</sup>	X <sup>1</sup>
Butyl phthalate, Di-n-	84-74-2	X <sup>1</sup>	X <sup>1</sup>
Chloroaniline, 4-	106-47-8	30	1
bis (2-Chloroethoxy)methane	111-91-1	X <sup>1</sup>	X <sup>1</sup>
bis (2- Chloroethyl)ether	111-44-4	30	0.7
bis (2-Chloroisopropyl) ether	108-60-1	30	0.7
Chloronaphthalene, 2-	91-58-7	X <sup>1</sup>	X <sup>1</sup>
Chlorophenol, 2-	95-57-8	10	0.7
Chrysene <sup>2</sup>	218-01-9	2	7
Dibenz(a,h)anthracene	53-70-3	0.5 <sup>2</sup>	0.7
Dibenzofuran	132-64-9	X <sup>1</sup>	X <sup>1</sup>
Dichlorobenzene, 1,2-	95-50-1	600	100
Dichlorobenzene, 1,3-	541-73-1	600	100



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Analyte	CASN	MCP CLEANUP STANDARDS	
		GW-1 (GW-3)	S-1/GW-1 (S-1/GW-3)
		µg/L (ppb)	µg/g (ppm)
<b>Table II B-2 Analyte List for SW-846 Method 8270C (Page 2 of 3)</b>			
Dichlorobenzene, 1,4-	106-46-7	5 <sup>2</sup>	2
Dichlorobenzidine, 3,3'-	91-94-1	80	1
Dichlorophenol, 2,4-	120-83-2	10	10
Diethyl phthalate	84-66-2	6,000/(30)	100/(0.7)
Dimethyl phthalate <sup>2</sup>	131-11-3	50,000/(30)	0.7
Dimethylphenol, 2,4-	105-67-9	100	0.7
Dinitrophenol, 2,4-	51-28-5	200	3
Dinitrotoluene, 2,4-	121-14-2	30	0.7
Dinitrotoluene, 2,6-	606-20-2	X <sup>1</sup>	X <sup>1</sup>
Dioxane, 1, 4	123-91-1	3.0	0.005 (see Note 5)
bis (2-Ethylhexyl) phthalate	117-81-7	6 <sup>2</sup>	100
Fluoranthene	206-44-0	100	600
Fluorene	86-73-7	300/(200)	400
Hexachlorobenzene	118-74-1	1 <sup>2</sup>	0.7
Hexachlorobutadiene	87-68-3	0.6 <sup>2</sup>	3
Hexachloroethane	67-72-1	8 <sup>2</sup>	6
Indeno (1,2,3-cd) pyrene	193-39-5	0.5 <sup>2</sup>	0.7
Isophorone	78-59-1	X <sup>1</sup>	X <sup>1</sup>
Methylnaphthalene, 2-	91-57-6	10	4
Methylphenol, 2 <sup>2</sup> -	95-48-7	X <sup>1</sup>	X <sup>1</sup>
Methylphenol, 3- <sup>2,4</sup>	108-39-4	X <sup>1</sup>	X <sup>1</sup>
Methylphenol, 4- <sup>2,4</sup>	106-44-5	X <sup>1</sup>	X <sup>1</sup>
Naphthalene	91-20-3	20	100
Nitrobenzene	98-95-3	X <sup>1</sup>	X <sup>1</sup>
Nitrophenol, 2- <sup>3</sup>	88-75-5	X <sup>1</sup>	X <sup>1</sup>
Nitrophenol, 4-	100-02-7	X <sup>1</sup>	X <sup>1</sup>



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Analyte	CASN	MCP CLEANUP STANDARDS	
		GW-1 (GW-3)	S-1/GW-1 (S-1/GW-3)
		µg/L (ppb)	µg/g (ppm)

**Table II B-2 Analyte List for SW-846 Method 8270C (Page 3 of 3)**

Octyl phthalate, di-n-	117-84-0	X <sup>1</sup>	X <sup>1</sup>
Pentachlorophenol	87-86-5	1 <sup>2</sup>	5
Phenanthrene	85-01-8	300/(50)	700/(100)
Phenol <sup>2</sup>	108-95-2	4,000	60
Pyrene	129-00-0	200	700
Trichlorobenzene, 1,2,4-	120-82-1	70	100
Trichlorophenol, 2,4,5-	95-95-4	200/(100)	3/(2)
Trichlorophenol, 2,4,6-	88-06-2	10	3

1. Department-Developed MCP Method 2 Standard. Use of these Standards is discretionary. See URL: <http://www.mass.gov/dep/bwsc/files/standard/method2/method2.htm>

2. Standard Reporting Limits for this compound may not be able to achieve regulatory compliance limit because of poor recovery or sensitivity when using GC/MS system in the "full scan" mode. A more effective extraction/sample introduction method or GC/MS – SIM (or some other more sensitive analytical procedure) may be required

3. Calibration Check Compound (CCC). See Section 7.4.5 and Table 4 of SW-846 Method 8270C

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

5. **This method is not approved for the analysis of soil and sediment samples for 1,4-Dioxane to support of MCP decisions.** The analysis of soil and sediment samples for 1,4-Dioxane in support of MCP response actions is described in MCP Analytical Method II A, SW-846 Method 8260B, Volatile Organics by GC/MS (Final, August 2004)



Title: : Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

## 2.0 Data Usability Assessment for SW-846 Method 8270C

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 Method 8270C which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document

## 3.0 Reporting Requirements for SW-846 Method 8270C

### 3.1 General Reporting Requirements for SW-846 Method 8270C

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

- Laboratory identification information presented in WSC-CAM-VII A, Section 2.4.1,
- Analytical results and supporting information in WSC-CAM-VII A, Section 2.4.2,
- Sample- and batch-specific QC information in WSC-CAM-VII A, Section 2.4.3,
- Laboratory Report Certification Statement in WSC-CAM-VII A, Section 2.4.4,
- Copy of the Analytical Report Certification Form in WSC-CAM-VII A, Exhibit VII A-1,
- Environmental Laboratory Case Narrative contents in WSC-CAM-VII A, Section 2.4.5,
- Chain of Custody Form requirements in WSC-CAM-VII A, Section 2.4.6

### 3.2 Specific Reporting Requirements for SW-846 Method 8270C

Specific Quality Control Requirements and Performance Standards for SW-846 Method 8270C are presented in Table II B-1. Specific reporting requirements for SW-846 Method 8270C are summarized below in Table II B-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis.



Title: : Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

**Table II B-3 Routine Reporting Requirements for SW-846 Method 8270C**

<b>Parameter</b>	<b>Required Analytical Deliverable</b>
GC/MS Tunes	<b>NO</b>
Initial Calibration	<b>NO</b>
Continuing Calibration (CCAL)	<b>NO</b>
Method (Preparation) Blank	<b>YES</b>
Laboratory Control Spikes (LCSs)	<b>YES</b>
LCS Duplicates	<b>YES</b>
Matrix Spike (MS)	<b>YES (if requested field MS)</b>
Matrix Spike Duplicate (MSD)	<b>YES (if requested field MSD)</b>
Field Matrix Duplicate (MD)	<b>YES (if requested by Data User)</b>
Surrogates	<b>YES</b>
Internal Standards (ISs)	<b>NO</b>
Tentatively Identified Compounds (TICs)	<b>YES (if requested by LSP)</b>
Identification and Quantification	<b>NO</b>
General Reporting Issues	<b>YES</b>

### 3.3 Tentatively Identified Compounds (TICs) by GC/MS

The evaluation of Tentatively Identified Compounds (TICs) in conjunction with GC/MS analyses (SW-846 Methods 8260B and 8270C) is a powerful and cost-effective analytical tool that can be utilized by the LSP to support MCP due diligence requirements. This analytical approach is particularly effective at 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:

**Always expected** when drinking water samples are analyzed,

**Not usually expected** at petroleum-only sites,

**Not usually expected** when the contaminants of concern have been previously identified,

**Not usually expected** when used to determine the extent and magnitude of contamination associated with a "known" release of OHM, and/or

**Should be considered**, at the discretion of the LSP, in support of site characterization activities for releases at locations with complex and/or uncertain history



Title: : Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

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

### 3.3.1 Reporting of Tentatively Identified Compounds (TICs)

If evaluated, all TICs that meet the chromatographic criteria presented in Section 1.0 of Appendix II B-2 must be reported by the laboratory either in the Environmental Laboratory Report or in the Environmental Laboratory's case narrative. In turn, the LSP must include a discussion regarding the disposition of all reported TICs as part of the MCP submission. 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 quality control 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-2 of this document.



Title: Sample Preservation, Container And Analytical Holding Time Specifications For Surface Water, Groundwater, Soil, And Sediment Matrices For Semivolatile Organic Compounds Analyzed In Support Of MCP Decision-Making

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for semivolatile organic compounds analyzed in support of MCP decision-making are summarized below and presented in Appendix VII-A of WSC-CAM-VIIA, "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)".

Matrix	Container <sup>1</sup>	Preservation <sup>2</sup>	Holding Time <sup>3</sup>
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to 4°C	7 days to extraction; 40 days from extraction to analysis <sup>3</sup>
Aqueous Samples, with Residual Chlorine <sup>4</sup>	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) <sup>5</sup> . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C	7 days to extraction; 40 days from extraction to analysis <sup>4</sup>
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap	Cool to 4°C	14 days to extraction; 40 days from extraction to analysis <sup>4</sup>
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a teflon-lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis <sup>4</sup>

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 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. Preparation or extraction must be commenced within 24 hours of thawing.

Temperature must never be allowed to go below – 20 °C to avoid damage to seals, etc.

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

4 SVOC samples extracts must be stored at – 10° C, protected from light, and stored in sealed vials (e .g., screw-cap or crimp-caped vials) with un-pierced PTFE-lined septa. See SW-846 Method 8270C, Section 6.1.

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.



Title: Guidance for Evaluation of Tentatively Identified Compounds (TICs)  
for SW-846 Method 8270C Under the MCP

A logic diagram for the Evaluation of Tentatively Identified Compounds (TICs) for SW-846 Method 8270C Under the MCP is presented in Exhibit II B-1. This exhibit graphically presents a systematic approach to evaluate tentatively identified compounds based on chromatographic, mass spectral, and toxic spectral characteristics criteria.

### 1.0 Chromatographic Criteria

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

### 1.1 Mass Spectral Criteria

- All spectra must be evaluated by a qualified mass spectrometrist.
- The spectral library match must be  $\geq 85\%$  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.
- The relative intensities of the major ions should agree within  $\pm 20\%$ .
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- 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).



Title: Guidance for Evaluation of Tentatively Identified Compounds (TICs)  
for SW-846 Method 8270C Under the MCP (*continued*)

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 analytical case narrative to support any “unknown” classification.

- TICs, which are identified as aliphatic petroleum hydrocarbons, do not have to be reported as TICs. However, there must be a statement in the case 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.

## 2.0 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

## 3.0 Reporting Criteria

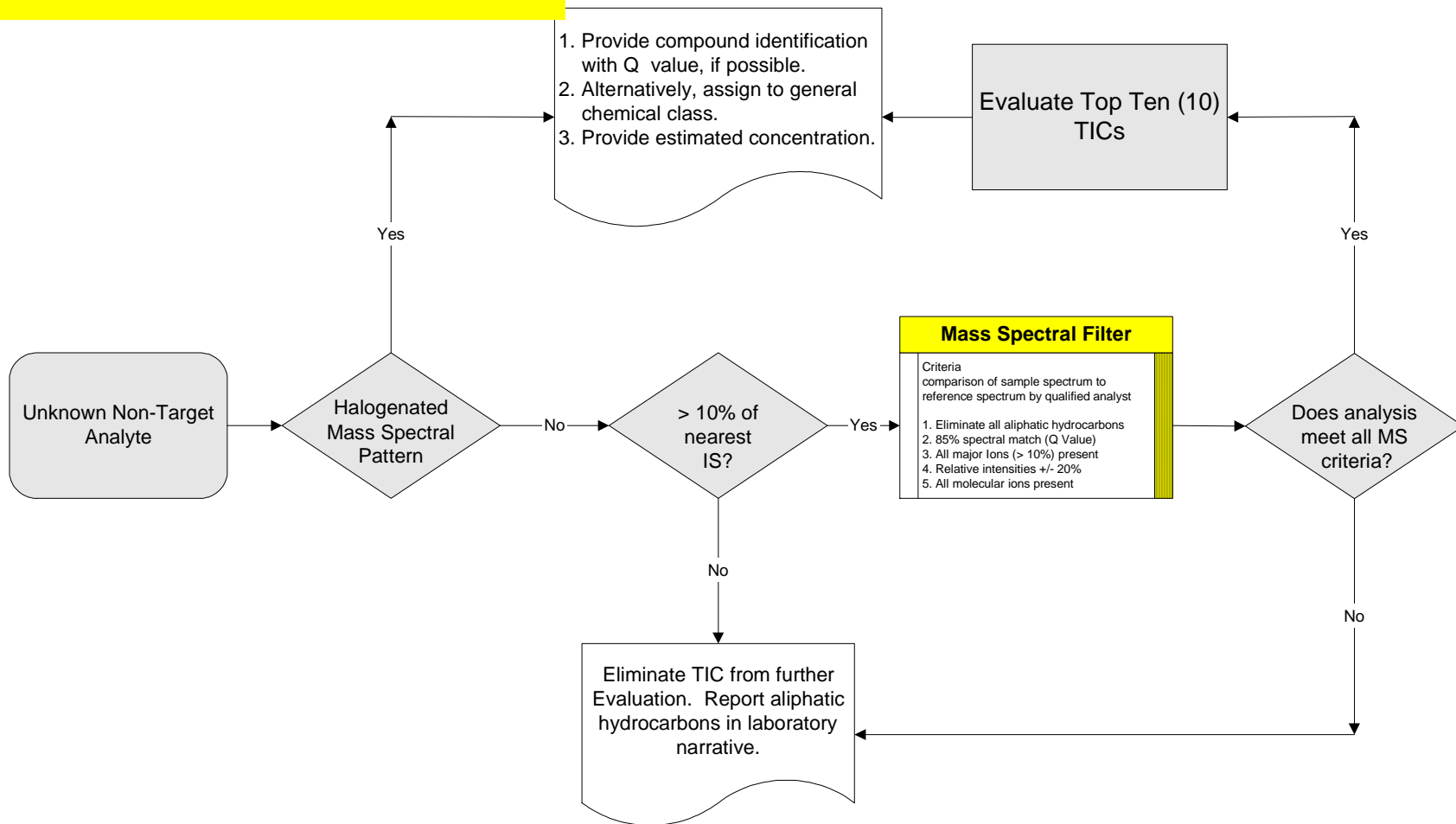
All TICs must be reported by the laboratory with the clear indication that the reported concentration is an estimated value unless analyte-specific calibration and QA/QC were performed as discussed in Section 3.3.1. This reporting requirement may be fulfilled by discussion in the analytical case narrative, by using a “J” flag designation, or by some other laboratory reporting convention to qualify the sample results. General environmental laboratory reporting recommendations are presented in CAM–VII A, Section 2.3.

A range of alternatives for LSPs to respond to reported TICs are presented in WSC-CAM – VII, Section 4.0. If an LSP 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 resampling. This contingency will require additional coordination and communication between the laboratory and the LSP.



Title: Guidance for Evaluation of Tentatively Identified Compounds (TICs) for SW-846 Method 8270C Under the MCP (*continued*)

**Analytical Note:** TICs need only be evaluated by the laboratory when specifically requested by the LSP





Title: 1,4-Dioxane Analytical Notes

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

1. Samples must be dechlorinated using sodium sulfite (50 mg/L) if residual chlorine is present and preserved with sodium bisulfate (approximately 1 g/L) at the time of sample collection. Samples must be maintained at 4<sup>0</sup> C until extracted. The sample must be extracted within 7 days of collection and analyzed within 40 days of the date of extraction.
2. Dichloromethane extracts are analyzed using modified SW-846 Method 8270 GC/MS-SIM. 1,4-Dioxane-d8 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-d4 is added post-extraction and is used to quantify 1,4-Dioxane-d8 as the method surrogate.
3. This method is only applicable for the analysis of groundwater and surface water samples and has an achievable reporting limit of 1ug/L, or lower. Large volume injection techniques may be employed to achieve even lower reporting limits, if required.
4. Samples (1000 ml) are extracted using chromatographic grade Methylene Chloride liquid-liquid extraction in a separatory funnel at a neutral pH in accordance with SW-846 method 3510. 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.
5. A known concentration of 1,4-Dioxane-d8 (1.0 ml) is added to the sample **prior** to liquid-liquid extraction as an internal standard (extraction standard) for quantification of 1,4-Dioxane using GC/MS-SIM. A known concentration of 1,4-Dichlorobenzene-d4 is added to the reduced-volume sample extract prior to analysis as an analytical surrogate.

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

<i>Compound</i>	<i>Primary Ion</i>	<i>Secondary Ion</i>
1,4-Dioxane	88	58, 43
1,4-Dioxane-d8	64	96
1,4-Dichlorobenzene-d4	152	115



Title: 1,4-Dioxane Analytical Notes (*continued*)

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	20% RSD.
Continuing Calibration	≤ 20 % Drift.
Relative Retention Times (RRT) of target analytes	Must be within ± 0.06 RRT units of the RRT of the standard component.
Area Counts (AC) of internal standard	Must be within –50 to +200% of the continuing calibration standard
Method Blank	No analyte > 0.5 ug/L
Surrogate Recovery	15% - 110 % Recovery
Laboratory Control Samples	40% - 140 % Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140 % Recovery; RPD ≤ 30

6. Data evaluation for 1,4-Dioxane includes the following:

- Intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- Relative retention time of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- Relative intensities of the characteristic ions 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 (proposed) incorporating GC/MS-SIM and SPE is currently under development. For Presumptive Certainty purposes, this method is considered a modification of SW-846 Method 8270C requiring a separate Initial Demonstration of Proficiency as described in the method.

1. Samples must be maintained at 4<sup>0</sup> C until extracted. The sample must be extracted within 7 days of collection and analyzed within 28 days of the date of extraction.
2. Solid Phase Extraction Tubes meeting the requirements US EPA Method 521 (Nitrosamines in Drinking Water) 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.



Title: 1,4-Dioxane Analytical Notes (*continued*)

3. Dichloromethane extracts are analyzed using modified SW-846 Method 8270 GC/MS-SIM with isotopic dilution (1,4-Dioxane-d8). The isotopically labeled compound, 1,4-Dioxane-d8, serves to correct the variability associated with extraction of the target analyte using this extraction procedure.
4. This method is only applicable for the analysis of groundwater and surface water samples and has an achievable reporting limit of 1ug/L, or lower. Large volume injection techniques may be employed to achieve even lower reporting limits, if required.
5. SPE tubes are installed on a standard SPE vacuum manifold from Supelco (Bellefonte, PA). SPE tubes containing two (2) grams of coconut charcoal (Restek, Bellefonte, PA) are cleaned and conditioned by sequential rinsing with three 5-mL aliquots of Methylene Chloride (DCM), 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 dry before the entire sample passes through the charcoal tube. A thin layer of methanol or water should remain on top of the charcoal.
6. A 500-mL water sample fortified with method analytes and the surrogate (1,4-dioxane-d8) is added to the top of the "wetted" sorbent and filtered through the charcoal at a rate of 5-10 mL/min using vacuum. After the entire sample volume is processed through the charcoal, a vacuum is pulled on the SPE tube for 5-10 min. The charcoal is then eluted with 10 mL DCM. The internal standard tetrahydrofuran (THF-d8) is added to the extract, and an aliquot of the extract is then transferred to a 2-mL auto-sampler vial. Because the extract contains some water, the extract must be dried with anhydrous sodium sulfate.
7. A 1-2  $\mu$ L sub-aliquot of the dried extract is then injected and analyzed by GC/MS using a 60 m X 0.25 mm DB-624 with a 1.4  $\mu$  film. Chromatographic conditions should be optimized for the analytical system.
8. The internal standard is used for 1,4-Dioxane quantification by GC/MS-SIM. A known concentration of THF-D8 is added to the sample extract prior to analysis as an analytical surrogate.

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

<i>Compound</i>	<i>Retention Time (min)</i>	<i>SIM Ions (m/z)</i>
1,4-Dioxane	8.85	58, <b>88</b> (TARGET)
1,4-Dioxane-d8	8.77	62, 64, <b>96</b> (SUR)
THF-d8	6.68	<b>46.</b> 78, 80 (IS)

(XX) m/z – Suggested quantification ion for TARGET, Surrogate and Internal Standard



Title: 1,4-Dioxane Analytical Notes (*continued*)

9. 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 SPE GC/MS-SIM method for "Presumptive Certainty" considerations.

Quality Control Criteria	Performance Standard
Initial Calibration	20% RSD.
Continuing Calibration	$\leq 20$ % Drift.
Relative Retention Times (RRT) of target analytes	Must be within $\pm 0.06$ RRT units of the RRT of the standard component.
Area Counts (AC) of internal standard	Must be within $-50$ to $+200\%$ of the continuing calibration standard
Method Blank	No analyte $> 0.5$ $\mu\text{g/L}$
Surrogate Recovery	15% - 110 % Recovery
Laboratory Control Samples	40% - 140 % Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140 % Recovery; RPD $\leq 30$

10. Data evaluation for 1,4-Dioxane includes the following:

- Intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other;
- Relative retention time of the sample component is within  $\pm 0.06$  RRT units of the RRT of the standard component; and
- Relative intensities of the characteristic ions agree within  $\pm 30\%$  of the relative intensities of these ions in the reference spectrum.