State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Semi-Volatile Organics by Method 8270, SW-846

Version 3.0

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Written by the Connecticut DEEP QA/QC Workgroup

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1.0	First version for Publication	7/11/05
2.0	Final version based upon public comments	July 2006
3.0	Updates to reflect CAM method updates to improve consistency between different states	May 2024

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Acronym List

ACRONYM	DEFINITION
AMU	Atomic mass units
ASE	Accelerated solvent extraction
CASN	Chemical Abstracts Service Number
CCV	Continuing calibration verification
%D	Percent difference or percent drift
DDT	Dichlorodiphenyltrichloroethane
DEEP	CT Department of Energy and Environmental Protection
DF	Dilution factor
DFTPP	Decafluorotriphenylphosphine
GC	Gas chromatograph
EP	Environmental Professional
GC/MS	Gas chromatography/mass spectrometry
ICV	Initial calibration verification
LCS	Laboratory control sample
mL	Milliliters
mL/min	milliliters per minute
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
OHM	Oil and Hazardous Materials
PAHs	Polycyclic aromatic hydrocarbons
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QC	Quality control
r/r^2	Correlation coefficient
RF	Response factor
RL	Reporting limit
RPD	Relative percent difference
%RSD	Percent relative standard deviation
RSRs	Remediation Standard Regulations
RT	Retention Time
SIM	Selective ion monitoring
SPE	Solid phase extraction
SVOCs	Semi-volatile organic compounds
UCM	Unresolved complex mixture
µg/kg	micrograms per kilogram
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µL	microliters
VOCs	Volatile organic compounds

1.0 Quality Assurance and Quality Control Requirements for Method 8270

1.1 Method Overview

SW-846 Method 8270 is gas chromatography/mass spectrometry ("GC/MS") procedure used to determine semi-volatile organic compounds ("SVOCs") in a variety of matrices including waters, soils, sediments, wastes, etc. This procedure requires an experienced GC/MS analyst familiar with the Quality Assurance and Quality Control ("QA/QC") requirements of the method. The sample introduction procedure requires the use of a solvent extraction procedure (See Table 1.0 for applicable extraction methods).

SW-846 Method 8270 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, organophosphorus pesticides, nitrosamines, haloethers, aldehydes, ethers, ketones, aromatic nitro compounds, and phenols.

In most cases, SW-846 Method 8270 is not appropriate for the quantitation of multi-component analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., or of single response chlorinated pesticides, because of limited sensitivity for these analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. For guidance on calibration and quantitation of these analytes refer to SW-846 Methods 8081 and 8082.

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 8270 for details.

All method references are to the latest promulgated version of the method found in *Test Methods for Evaluating Solid Waste, SW-846*.

1.2 Summary of Method 8270

1.2.1 Sample Extraction and Cleanup

Samples for analysis by Method 8270 require extraction by one of the following methods listed in Table 1.0 below:

Table 1.0: Extraction Methods

SW-846 Method	Matrix	Description
3535	Aqueous	Solid-Phase Extraction ("SPE")
3510	Aqueous	Separatory Funnel Liquid-Liquid Extraction
3520	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3540	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545	Soil/Sediment	Pressurized Fluid Extraction ("PFE")
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction ("MSE")
3550	Contaminated Solids[1]	Ultrasonic Extraction
3580	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.		

In very limited applications, direct injection of an aqueous sample into the GC/MS system with a 10-µL syringe may be appropriate. The reporting limit 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 listed in Table 2.0 prior to GC/MS analysis by SW-846 Method 8270.

Table 2.0: Clean-up Methods

Analytes of Interest	Cleanup Methods
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

1.2.2 GC/MS Analysis in Full Scan Mode

The SVOCs are extracted from the sample using the appropriate method (Table 1.0). The solvent extract is concentrated and then aliquots of the concentrate are injected into the gas chromatograph (“GC”). The analytes are then introduced onto a capillary column for analysis. The GC oven is temperature programmed to facilitate separation of the analytes which are then detected by a mass spectrometer which is interfaced to the GC. In a full scan operational mode, 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.

Identification of target analytes is accomplished by comparing the retention time and electron impact mass spectra of the analytes to that of a standard analyzed under the same conditions. Quantitation is accomplished by using the response of a major (quantitation) ion relative to an internal standard and a response factor generated from a calibration curve consisting of a minimum of five points, or six if non-linear regression is used.

1.2.3 GC/MS System Operating in the Selective Ion Monitoring (“SIM”) Mode

A GC/MS system is generally operated in the Selective Ion Monitoring (“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. Coupled with full scan data, positive identification of analytes of concern is preserved. 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, and analyte quantification are the same whether the GC/MS system is operated in the full scan or SIM mode. Use of the SIM Mode may require different internal standards and surrogates from the full scan mode. A library search for tentatively identified compounds is not possible when an instrument is operated in the SIM Mode.

1.3 Method Interferences

Refer to SW-846 Method 8270 for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination.

1.3.1 Chemical Contaminants

Major contaminant sources for Method 8270 include, but are not limited to, plastics, impurities in laboratory chemicals, contaminated laboratory ware, etc. The use of non-polytetrafluoroethylene ("PTFE") thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials may contaminate the analytical system.

Analysis of blanks provides information about the presence of contaminants. When potential interfering peaks or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the contamination and eliminate it. **Subtracting blank values from sample results is not permitted.** Any method blank exceedances should be fully documented in the laboratory report narrative.

1.3.2 Cross-contamination/ Carryover

Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of SVOCs (ghost peaks). Syringes on the autosampler may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of solvent blanks. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run.

1.4 Quality Control Requirements for SW-846 Method 8270

1.4.1 Reporting Limits/Lower Limit of Quantitation for Method 8270

The reporting limit ("RL"), or lower limit of quantitation ("LLOQ"), for a compound is dependent on the concentration of the lowest non-zero standard in the initial calibration, sample weight/volume, extraction procedure, and moisture content. Table 3.0 lists approximate reporting limits for various matrices utilizing the standard quadrupole mass spectrometer. Solid matrices in this table assume 100% solids.

Table 3.0: Reporting Limits / Lower Limits of Quantitation[1]

Matrix	Typical Reporting Limit
Water	10 µg/L
Soil, Low Level	330 µg/Kg
Soil, High Level	10,000 µg/Kg

[1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.

Lower limits may be achieved using select ion monitoring, an ion trap mass spectrometer, or newer instrumentation. Certain analytes, notably water-soluble compounds such as 1,4-Dioxane, have poor extraction efficiencies. This will mandate higher calibration levels for these type compounds and therefore higher RLs/LLOQs.

Moisture content of soils and sediments will also raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RLs/LLOQs to be raised. 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 to meet the project Data Quality Objectives ("DQOs"). To meet the RLs/LLOQs applicable to project DQOs, it may be necessary to modify the analytical method by using increased sample volume or mass or employing selective ion monitoring. In such cases the modifications must be noted in the laboratory report narrative.

1.4.2 General Quality Control Requirements

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 for semi-volatile organics.

Refer to SW-846 Method 8000 for general Quality Control ("QC") requirements for all chromatographic methods, including SW-846 Method 8270. These requirements ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all chromatographic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the GC system operation may be found in SW-846 Method 8000. Instrument QC and method performance requirements for the GC/MS system may be found in SW-846 Method 8270.

The minimum requirements for a formal QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and LCS duplicates ("LCSD") to assess precision. The use of site-specific matrix spikes and matrix spike duplicates ("MSD") is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Field, rinsate, or other blanks should not be used for MS/MD's.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative method being used. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform this method. These data must meet or fall within the performance standards as presented in Section 1.4 and Table 1A of this RCP and presented in SW-846 Method 8000. The IDOC must include the following elements provided in Table 4.0:

Table 4.0: IDOC Requirements

QC Element	Performance Criteria
Decafluorotriphenylphosphine ("DFTPP") Tuning	See Method 8270
Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Average Recovery	Table 1A [SW-846 Method 8000]
% Relative Standard Deviation	Table 1A [SW-846 Method 8000]
Surrogate Recovery	Table 1A
Internal Standards	Table 1A

Because of the extensive analyte list and number of QC elements associated with the IDOC, it should be expected that one or more analytes may not meet the performance standards for one or more QC elements. The laboratory should make every effort to find and correct the problem and repeat the analysis. All non-conforming analytes along with the laboratory acceptance criteria should be noted in the IDOC data. This information should be kept on-file at the laboratory.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and relative percent difference (“RPD”) limits, and surrogate recovery limits. These limits must be equal to or fall within the limits specified in Table 1A.

1.4.3 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8270

Specific QA/QC requirements and performance standards for SW-846 Method 8270 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with “Reasonable Confidence” regarding the usability of analytical data to support environmental decisions. The concept of "Reasonable Confidence" is explained on the CT Department of Energy and Environmental Protection (“DEEP”) website.

While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally, accept "Reasonable Confidence" data. In order to achieve “Reasonable Confidence” parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming analytical performance standards; and

3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

Table 1A: Specific QA/QC Requirements and Performance Standards for Method 8270

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Capability ("IDOC")	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 SW-846 8000.	No	Refer to SW-846 8000 and Section 1.4.2 of this protocol.	NA
GC/MS Tunes with DFTPP	Inter-Laboratory Consistency & Comparability	(1) Criteria listed in criteria for DFTPP listed in SW-846 8270. (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. (5) If using a single quadrupole mass spectrometer then utilize tuning parameters it items 1, 2, and 3 above. If using a triple quadrupole mass spectrometer then refer to EPA method 8270. 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. Note: Tune 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.	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 factor non-conformances in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration ("ICAL")	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when ICV or CCV does not meet the performance standards, and when major instrument maintenance is performed (see Method 8000 for guidance). (2) Minimum of 5 standards (or 6 if non-linear regression used)[1]. (3) Low standard must be ≤ RL/LLOQ. (4) %RSD ≤20, r ≥0.99 (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 for each compound as per SW-846 8270 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) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve, recoveries must be 50-150%. (10) 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. (11) If regression is used, must not be forced through the origin.	No	(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: (a) the RL/LLOQ limit must be reported as an estimated value[1], or (b) 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.	(1) Sample analysis cannot proceed without a valid initial calibration. (2) Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory report narrative. If non-linear regression is used for calibration (e.g., quadratic equation), this must be noted in the laboratory report narrative with a list of compounds affected.
Initial Calibration Verification ("ICV")	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[2] which must exhibit percent recoveries between 40-160%.	No	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[2], report non-conforming compounds in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Verification ("CCV")	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 ≤20 for each target analyte. (5) Minimum RFs as per SW-846 8270. (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.	No	(1) Recalibrate if >20% 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 reanalyze the continuing calibration. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D <40.	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory report narrative.
Method Blank ("MB")	Laboratory Method Sensitivity & Contamination Evaluation	(1) Extracted every ≤20 field samples or every batch, whichever is more frequent, prior to sample analysis and after calibration standards (2) Matrix specific (e.g., water, soil). (3) Target analytes must be <RL/LLOQ except for common laboratory contaminants (phthalates) which must be <3x the RL/LLOQ.	Yes	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and reanalyze 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 report narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time, the lab must report results of both the initial extraction and re-extraction.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample ("LCS")	Laboratory Method Accuracy	(1) Extract with every batch or every ≤20 field samples, whichever is more frequent. (2) Concentration level near midpoint curve. (3) Must contain all target analytes. (4) Matrix specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes[2] which must exhibit percent recoveries between 15-140%. (6) Must be prepared in water-miscible solvent (e.g., acetone, methanol).	Yes	(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 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 compounds 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 report narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes[2], report non-conforming compounds laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the lab must report results of both the initial extraction and re-extraction.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
LCS Duplicate ("LCSD")	Laboratory Method Accuracy & Precision	(1) Extracted with every batch or every ≤20 field samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix-specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes[2] which must exhibit percent recoveries between 15-140%. (6) RPD must be ≤20% for waters and ≤30% for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	(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 report narrative. (2) If recovery is outside of 40-140% for a base-neutral compound or 30-130% for any acid compound, including "difficult" analytes[2] or if RPD is outside of criteria, report non-conforming compounds in laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the lab must report results of both the initial extraction and re-extraction.
Site-Specific Matrix Spike / Matrix Spike Duplicate ("MS/MSD")	Method Precision and Accuracy in Sample Matrix	(1) Every ≤20 field samples (selected at discretion of lab or at request of data user). (2) Matrix-specific (e.g., aqueous, soil). (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries between 40-140% for base-neutral compounds and between 30-130% for the acid compounds (6) RPDs ≤20% for waters and ≤30% for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol). (8) Field blanks, trip blanks, etc. cannot be used for MS/MSDs.	Yes ONLY when requested by data user	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note non-conformances in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
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, p-terphenyl-d14. Recommended acid surrogates: phenol-d5, 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) Soil percent recoveries within 30-130%. (3) Water percent recoveries within 30-130% for base-neutrals, 15-110% for acidic compounds.	Yes	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% the following applies: (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. (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 with dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, re-extraction and/or reanalysis is not required.	(1) Report non-conformances in laboratory report narrative. (2) If re-extraction yields similar surrogate non-conformances, the lab must report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries the lab 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 lab 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.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Internal Standards	Laboratory Analytical Accuracy & Method Accuracy in Sample Matrix	(1) Minimum of 6 at retention time across GC Run. NOTE: For SIM analyses, the number of internal standards will vary depending on number of 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	If any internal standards are outside of limits, reanalyze sample extract 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 non-conformances in laboratory report 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 lab must report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the lab 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 lab must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the lab must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The lab must use the average RF, 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) Do no report concentrations below the RL/LLOQ.	NA	NA	If the average RF or linear regression not used for quantitation (e.g., quadratic equation), it must be noted in laboratory report narrative with list of affected analytes. Quadratic or polynomial fits require 6 & 7 calibration points.
Analyte Identification	NA	Refer to SW-846 8270.	NA	NA	NA

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
General Reporting Issues	NA	(1) Do not report concentrations below the RL/LLOQ. If reporting estimated concentrations below the RL/LLOQ, labs must indicate that RCP was not followed. The lab must report results for samples and blanks in a consistent manner. (2) Concentrations below the RL/LLOQ should be reported as "ND" with the sample-specific RL/LLOQ also reported. (3) Dilutions: if diluted and undiluted analyses are performed, the lab 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. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to RSR regulatory standards. (5) Refer to Table 6.0 for requirements regarding preservation, cooler temp, and holding time.	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 documented in laboratory report narrative and must be maintained in laboratory records. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory report narrative. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix A-2.9. (4) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report narrative. (5) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative.

[1]Six standards are required for a quadratic equation calibration curve, and seven are required for a polynomial fit. In either case the correlation coefficient must be ≥ 0.990. Surrogates may be calibrated using a single point, at the same concentration as added to all samples, blanks, etc.

[2]Potentially Difficult Classes of Compounds include: Analines, Phenols, Phthalates, plus potentially difficult compounds including Hexachlorocyclopentadiene, Pyridine, and others (See EPA Methods 8000 and 8270 for other compounds).

If the RL/LLOQ is estimated due to unacceptable recovery of the lowest standard, the RL/LLOQ has not been achieved; Question 5b of the "Reasonable Confidence Protocol Laboratory Analysis QA/QC Certification Form" must be answered "NO" and this must be addressed in the laboratory report narrative.

1.5 Special Analytical Considerations for SW-846 Method 8270

Because of the variable solubility, extraction efficiency and analytical sensitivity of the different classes of SVOCs that are potentially analyzable by SW-846 Method 8270, the recovery ranges presented in Table 1A 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:

- 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%).

- Semi-volatile 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%).

- If 1,4-dioxane is a contaminant of concern for the site, special analytical techniques, as listed below, must be utilized.

  As described in Appendix B, 1,4-dioxane in groundwater/surface water may be analyzed using GC/MS-SIM with isotopic dilution using 1,4-dioxane-d8 as an 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 8260 is an acceptable approach. However, if elevated concentrations of other chlorinated 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.

In these examples, the EP 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; the EP 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 1A.

1.6 Analyte List for SW-846 Method 8270

The DEEP analyte list for SW-846 Method 8270 is presented in Table 1B. The compounds listed are readily determined by Method 8270. Most of the compounds listed have Connecticut RSR Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

Table 1B: Analyte List For SW-846 Method 8270

Analyte	CASN[1]	Analyte	CASN[1]
Acenaphthene	83329	2,4-Dinitrophenol	51285
Acenaphthylene	208968	2,4-Dinitrotoluene	121142
Acetophenone	98862	2,6-Dinitrotoluene	606202
Aniline	62533	Di-n-octylphthalate	117840
Anthracene	120127	1,4-Dioxane[2]	123911
Azobenzene	103333	Fluoranthene	206440
Benzo(a)anthracene	56553	Fluorene	86737
Benzo(b)fluoranthene	205992	Hexachlorobenzene	118741
Benzo(g,h,i)perylene	191242	Hexachlorobutadiene	87683
Benzo(k)fluoranthene	207089	Hexachlorocyclopentadiene	77474
Benzo(a)pyrene	50328	Hexachloroethane	67721
Bis(2-chloroethyl)ether	111444	Indeno(1,2,3-cd)pyrene	193395
Bis(2-chloroethoxy)methane	111911	Isophorone	78591
Bis(2-chloroisopropyl)ether[3]	108601	2-Methylnaphthalene	91576
Bis(2-ethylhexyl)phthalate	117817	2-Methylphenol (o-Cresol)	95487
4-Bromophenyl-phenylether	101553	3-Methylphenol	108394
Butylbenzylphthalate	85687	4-Methylphenol (p-Cresol)[4]	106445
Carbazole	86748	Naphthalene	91203
4-Chloroaniline	106478	2-Nitroaniline	88744
4-Chloro-3-methylphenol	59507	3-Nitroaniline	99092
2-Chloronaphthalene	91587	4-Nitroaniline	100016
2-Chlorophenol	95578	Nitrobenzene	98953
4-Chlorophenyl-phenylether	7005723	2-Nitrophenol	88755
Chrysene	218019	4-Nitrophenol	100027
Dibenzofuran	132649	N-nitrosodiphenylamine	86306
Dibenzo(a,h)anthracene	53703	N-Nitroso-di-n-propylamine	621647
1,2-Dichlorobenzene	95501	Pentachloronitrobenzene	82688
1,3-Dichlorobenzene	541731	Pentachlorophenol	87865
1,4-Dichlorobenzene	106467	Phenanthrene	85018
3,3'-Dichlorobenzidine	91941	Phenol	108952
2,4-Dichlorophenol	120832	Pyrene	129000
Diethylphthalate	84662	Pyridine	110861
2,4-Dimethylphenol	105679	1,2,4,5-Tetrachlorobenzene	95943
Dimethylphthalate	131113	1,2,4-Trichlorobenzene	120821
Di-n-butylphthalate	84742	2,4,5-Trichlorophenol	95954
4,6-Dinitro-2-methylphenol	534521	2,4,6-Trichlorophenol	88062

[1] CASN – Chemical Abstract Service Number
[2] These compounds may require analysis by an alternative method to achieve applicable RSR criteria.
[3] Also known as 2,2'oxybis(1-chloropropane)
[4] 3-Methylphenol and 4-Methylphenol cannot be separated chromatographically, and are calibrated and reported as any combination of the two isomers.

1.6.1 Additional Reporting Requirements for SW-846 Method 8270

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEEP strongly recommends that full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.7 Routine Reporting Deliverables for Method 8270

The following table (Table 5.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report these items in special circumstances.

Table 5.0: Report Deliverables

Parameter	Deliverable	Comments
GC/MS Tunes	NO	Note non-conformances in laboratory report narrative
Initial Calibration	NO	Note non-conformances in laboratory report narrative
Continuing Calibration	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive results above RL with “B” flag.
Lab Control Sample/Lab Control Sample Duplicate	YES	Note non-conformances in laboratory report narrative
Site Specific Matrix Spike/ Matrix Spike Duplicate	YES (If requested by data user)	Note non-conformances in laboratory report narrative
Surrogate Recoveries	YES	Note non-conformances in laboratory report narrative
Internal Standard Areas	NO	Note non-conformances in laboratory report narrative
Tentatively Identified Compounds	YES (If requested by data user)	Flag all values as estimated (“J” Flag)
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Chain of Custody Form	YES	Signed by sample collector, courier, and laboratory

1.7.1 Reporting and Flagging of Results

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as “ND” (Not Detected at the Specified RL/LLOQ). The RL/LLOQ for each compound in each sample must be listed on the report and consider the exact sample mass, any dilution factors, percent moisture, etc.
- Compounds detected above the RL/LLOQ in blanks and found in samples, also above the RL/LLOQ, shall be flagged with a “B” suffix (e.g., 25B).
- Report results for any library search compounds as estimated using a “J” suffix (e.g., 25J).
- All soil/sediment results shall be reported on a dry weight basis.

1.8 Sample Containers, Preservation, and Holding Times

Table 6.0 identifies the type of containers, preservation requirements, and holding times dependent upon analyte and matrix.

Table 6.0: Sample Containers, Preservations and Holding Times

Matrix	Container[1]	Preservative[2]	Holding Time
Aqueous with no chlorine present	1-liter amber glass bottle with Teflon line cap	Store at 4 ± 2º C, but not frozen.	7 days to extraction. 40 days from extraction to analysis.
Aqueous with chlorine present	1-liter amber glass bottle with Teflon line cap	Neutralize chlorine with either 25 mg ascorbic acid or 3 mg sodium thiosulfate. Store at 4 ± 2º C, but not frozen.	7 days to extraction. 40 days from extraction to analysis.
Soil/Sediment samples.	250 mL amber glass jar with Teflon lined cap.	Cool to 4 ± 2º C.	14 days to extraction. 40 days from extraction to analysis. Up to one year for samples frozen within 48 hours of collection.[3]
High Concentration Waste Samples	Collect in amber glass jar with Teflon lined cap.	Cool 4 ± 2º C.	14 days to extraction. 40 days from extraction to analysis.

[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]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.

[3]If the freezing option is selected, the sample must be frozen within 48 hours of collection. The holding time recommences when thawing begins. The total holding time is calculated from the time of collection to freezing plus the time allowed for thawing. The total elapsed time must be less than 14 days.

1.9 Tentatively Identified Compounds

The evaluation of Tentatively Identified Compounds ("TICs") in conjunction with GC/MS analyses is a powerful and cost-effective analytical tool that can be utilized by EP to support RSR 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 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 Oil and Hazardous Materials ("OHM"), and/or
- **Should be considered**, at the discretion of the EP, in support of site characterization activities for releases at locations with complex and/or uncertain history

It should be noted that TICs only need to be evaluated by the laboratory for drinking water or when specifically requested by the EP.

*Meaning water directly consumed from either public or private water supplies.

1.9.1 Reporting of Tentatively Identified Compounds ("TICs")

If evaluated, all TICs that meet the chromatographic criteria presented in Appendix A of this RCP must be reported by the laboratory either in the Environmental Laboratory Report or in the laboratory report narrative. In turn, the EP must include a discussion regarding the disposition of all reported TICs as part of the RSR submission to DEEP. 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. No regulatory judgments or remedial decisions should be made without re-analysis of samples for the TICs using a five-point analyte specific calibration and appropriate quality control. This may also require re-sampling to meet analytical holding times.

Appendix A: Laboratory Requirements for Evaluation of Tentatively Identified Compounds Method 8270

A-1. Chromatographic Criteria

A-1.1 Initially include all the non-target compounds that have a peak area count ≥10% of the nearest internal standard. The EP may request evaluation of unknown peaks before the first internal standard based on site-specific information.

A-2. Mass Spectral Criteria

A-2.1 All spectra must be evaluated by a qualified mass spectrometrist and the Organic Supervisor/Laboratory Director.

A-2.2 The spectral library match must be ≥ 85% for a tentative identification to be made.

A-2.3 The major ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample spectrum.

A-2.4 The relative intensities of the major ions must agree within ± 20%.

A-2.5 Molecular ions present in the reference spectrum should be present in the sample spectrum.

A-2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks.

A-2.7 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).

A-2.8 Spectra identified as “unknown” should be assigned to a general chemical class, if possible. Classification as a halogenated hydrocarbon, aldehydes/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 laboratory report narrative to support any “unknown” classification.

A-2.9 TICs which are identified as petroleum aliphatic hydrocarbons should not be reported as TICs. TICs identified as aromatics or other hydrocarbons should be reported. However, there must be a statement in the laboratory report narrative discussing the presence of these hydrocarbons in the sample(s).

A-2.10 Aldol condensation products are formed when acetone is used as an extraction solvent. Two common aldol condensation products are mesityl oxide (or 4-methyl-3-pentene-2-one) and diacetone alcohol (or 4-methyl-4-hydroxy-2-pentanone). Aldol condensation products, if present, should be reported as “Aldol Condensation Products”, but not counted as part of the top 20 TICs and flagged with an “A” suffix.

A-2.11 It has been found that under certain conditions isophorone can be formed when using acetone with the accelerated solvent extraction (ASE) method. Laboratories are cautioned to investigate when high concentrations of isophorone are present and samples have been extracted using this technique. This reaction seems more prevalent if the sample has a high pH such as might be found when the sample contains concrete.

A-2.12 After the above criteria are met, the top twenty (20) compounds for SVOCs, chosen by comparing the area of the TIC to the area of the nearest internal standard, must be tentatively identified, quantitated, and reported. All TIC concentrations should be flagged as estimated by using a “J” suffix.

A-3. Toxic Spectral Characteristics Criteria

A-3.1 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.

A-4. Semi-Quantitative Analysis

A-4.1 Once a TIC has been identified, the semi-quantitation of that compound will be based on the integrated abundance of the TIC and internal standard total ion chromatogram. The response factor for all TICs will be assumed to be 1.0. The internal standard used shall be the one with the nearest retention time to a given TIC and that is interference free.

A-4.2 The resulting semi-quantitative concentration must be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine the concentration.

A-5. Reporting Criteria

A-5.1 All TICs eluting after the first target compound and 3 minutes after the last target compound meeting the requirements in A-2 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. This reporting requirement may be fulfilled by discussion in the laboratory report narrative or by using a “J” flag designation.

NOTE: In most circumstances the laboratory must order standards to be able to run a calibration curve and the appropriate QA/QC. The EP should be prepared to expect longer analytical turn-around-times in order to attain TIC results that are scientifically defensible.

Appendix B: Analytical Notes for 1,4-Dioxane Analysis

B.1 SW-846 Method 3510 – Liquid-Liquid Extraction ("LLE")

B-1.1 Methylene chloride 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.

B-1.2 This method is only applicable for the analysis of groundwater and surface water samples and has an achievable reporting limit of 1 µg/L, or lower. Large volume injection techniques may be employed to achieve even lower reporting limits, if required.

B-1.3 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.

B-1.4 A known concentration of 1,4-Dioxane-d8 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 internal standard.

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

Table B-1: Suggested Ions of LLE

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 1A also apply to the analysis of groundwater and surface water samples using this modified LLE GC/MS-SIM method for "Reasonable Confidence" considerations.

Table B-2: Quality Control Criteria and Performance Standards for LLE

Quality Control Criteria	Performance Standard
Initial Calibration	% RSD ≤20 or r ≥ 0.99.
Continuing Calibration	%D ≤ 20.
Relative Retention Times (RRT) of target analytes	Must within ± 0.06 RRT units of the RRT of the standard component.
Area Counts of internal standard	Must be within –50 to +200% of the continuing calibration standard
Method Blank	No analyte > 0.5 µg/L
Surrogate Recovery	15% - 110% Recovery
Laboratory Control Samples	40% - 140% Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140% Recovery; RPD ≤ 30

B-1.5 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.

- Relative retention time of the sample component must be within ± 0.06 RRT units of the RRT of the standard component.

- Relative intensities of the characteristic ions must agree within ± 30% of the relative intensities of these ions in the reference spectrum.

B-2. 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 Reasonable Confidence purposes, this method is considered a modification of SW-846 Method 8270, requiring a separate Initial Demonstration of Capability as described in the method.

B-2.1 Solid Phase Extraction Tubes meeting the requirements US 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 (Restek, Bellefonte, PA) and a tube volume of 6 mL.

B-2.2 Methylene chloride 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.

B-2.3 This method is only applicable for the analysis of groundwater and surface water samples and has an achievable reporting limit of 1 µg/L, or lower. Large volume injection techniques may be employed to achieve even lower reporting limits, if required.

B-2.4 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 dry before the entire sample passes through the charcoal tube. A thin layer of methanol or water should remain on top of the charcoal.

B-2.5 A 500-mL water sample fortified with 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 minutes. The charcoal is then eluted with 10 mL of methylene chloride. The internal standard tetrahydrofuran-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.

B-2. 6 A 1-2 µL 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 µ film. Chromatographic conditions should be optimized for the analytical system.

B-2.7 The internal standard is used for 1,4-Dioxane quantification by GC/MS-SIM. A known concentration of tetrahydrofuran-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:

Table B-3: Suggested Ions for SPE

Compound	Primary Ion	Secondary Ion
1,4-Dioxane	88	58, 43
1,4-Dioxane-d8	64	96
Tetrahydrofuran-d8	46	78, 80

B-2.8 Unless otherwise specified in the following table, all Quality Control and Performance Standards specified in Table 1A also apply to the analysis of groundwater and surface water samples using this SPE GC/MS-SIM method for "Reasonable Confidence" considerations.

Table B-4: Quality Control Criteria and Performance Standards for SPE

Quality Control Criteria	Performance Standard
Initial Calibration	% RSD ≤20 or r ≥ 0.99.
Continuing Calibration	%D ≤ 20.
Relative Retention Times ("RRT") of target analytes	Must within ± 0.06 RRT units of the RRT of the standard component.
Area Counts of internal standard	Must be within –50 to +200% of the continuing calibration standard
Method Blank	No analyte > 0.5 *µg*/L
Surrogate Recovery	15% - 110% Recovery
Laboratory Control Samples	40% - 140% Recovery
Matrix Spike/Matrix Spike Duplicate	30% - 140% Recovery; RPD ≤ 30

B-2.9 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;
- Relative retention time of the sample component must be within ± 0.06 RRT units of the RRT of the standard component; and
- Relative intensities of the characteristic ions must agree within ± 30% of the relative intensities of these ions in the reference spectrum.