State Of Connecticut
Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Semivolatile Organics by Method 8270, SW-846

Version 2.0
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Written by the Connecticut DEP QA/QC Workgroup

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1	First version for Publication	7/11/05
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1.0 QA/QC Requirements for Method 8270

1.1 Method Overview

Method 8270 is gas chromatography/mass spectrometry procedure used to determine semivolatile organic compounds (SVOC's) in a variety of matrices including waters, soils, sediments, wastes, etc. This procedure requires an experienced GC/MS analyst familiar with the QA/QC requirements of the method. The sample introduction procedure requires the use of a solvent extraction procedure. All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, 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, 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. Refer to Sec. 7.0 of SW-846 Methods 8081A and 8082 for guidance on calibration and quantitation of these analytes.

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 Method 8270, Section 1.4 for details.

1.1.1 Reporting Limits for Method 8270

The reporting limit (RL) for a compound is dependent on the concentration of the lowest standard in the initial calibration, sample weight/volume, extraction procedure, and moisture content. The following table lists approximate reporting limits for various matrices utilizing the standard quadrapole mass spectrometer. Solid matrices in this table assume 100% solids.

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 RL's.

Table 1.0 Typical Reporting Limits

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

Moisture content of soils and sediments will also raise the RL, 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 RL's to be raised.

Sample container type, preservation requirements, and holding times for waters, soils, and sediments are presented in Table 2A of this document.

1.1.2 General Quality Control Requirements

Each laboratory is required to operate a formal quality assurance program and be certified by the Connecticut Department of Public Health for the analysis performed. The minimum requirements include initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples (LCS) to assess precision and accuracy. The use of site specific matrix spikes and matrix spike duplicates is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making good decisions.

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 1.5 and Table 1A. See Section 8.4 of Method 8000 in SW-846 for the procedure. The Initial Demonstration of Proficiency must include the following elements:

Table 1.1 IDOC Requirements

QC Element	Performance Criteria
DFTPP Tuning	Table 1C
Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Average Recovery	Table 1A
% Relative Standard Deviation	Table 1A
Surrogate Recovery	Table 1A
Internal Standards	Table 1A

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 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 Initial Demonstration of Proficiency data.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and precision (RPD) limits, and surrogate recovery limits. These limits must meet or exceed the limits specified in Table 1A.

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:

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 [1]	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

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

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

Analytes of Interest	Cleanup Methods
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 semivolatile organic compounds are extracted from the sample using the appropriate method. The solvent extract is concentrated and then aliquots are injected into the gas chromatograph. The analytes are then introduced onto a capillary column for analysis. The gas chromatograph (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 five-point curve.

1.2.3 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. Use of the SIM Mode may require different internal standards and surrogates from the 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

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 exceedences 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 SVOC's (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 General Quality Control Requirements for Determinative Chromatography Methods

Refer to SW-846 Method 8000 for general quality control requirements for all chromatographic methods, including SW-846 Method 8270. These requirements insure 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 8000, Section 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 8270, Sections 8.0 and 9.0, respectively.

1.4.2 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 environmental professional (EP) with "Reasonable Confidence" regarding the usability of analytical data to support DEP decisions.

While optional, parties electing to utilize these protocols will be assured that "Reasonable Confidence" data, will be generally accepted by agency reviewers. 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, as necessary, compliance with performance standards prescribed in Table 1A for this test method; and

3. Adopt the reporting formats and elements specified in Section 1.7 of this method.

1.4.3 Site Specific Matrix Spike (MS), Matrix Spike Duplicate (MSD) Samples

It is strongly recommended that site specific MS/MSD samples be analyzed from each site, and each matrix type sampled. Percent recovery data from site specific samples allow 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 and are in general discouraged. Non-site specific MS/MSD's should not be reported for the RCP's. Additionally trip blanks, field blanks, rinsate blanks, etc. should not be used for MS/MSD's.

1.4.4 Special Analytical Considerations for SW-846 Method 8270

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 1A 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%).

- 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%).

In both of 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.

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

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 listed in Table 1C of this document. (the same criteria must be used for all analyses) 2) Every 12 hours 3) 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 Analysis	NO	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified. Report peak tailing excedences in narrative
Initial Calibration (ICAL)	Laboratory Analytical Accuracy	1) Minimum of 5 standards. (Note 1) 2) Low standard must be ≤ reporting limit (RL) 3)Full Scan % RSD ≤ 15 or "r" ≥ 0.990 for all compounds except CCC's, which must be ≤ 30% RSD or "r" ≥ 0.990. SIM % RSD ≤30 or "r" ≥ 0.990. 4) Must contain all target analytes 5) If regression is used, must not be forced through the origin. 6) If SIM is used, laboratory must monitor at least two ions/analyte for all targets, surrogates, and IS's. 7) Minimum RF for all compounds > 0.05.	NO	Recalibrate as required by method (1) if any of CCC %RSDs or if any one of CCC "r" <0.990 or (2) if >20% of remaining analytes have %RSD >30 or "r" < 0.990.	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 quantitation (e.g. use of a quadratic equation), this must be noted in narrative with a list of affected analytes.
ICAL Verification Standard	Laboratory Analytical Accuracy	1) Each ICAL must be verified against a second source standard. 2) Std should be at mid-point 3) All target analytes present	NO	1) Compounds must recover within 80-120% 2) Laboratories are allowed to have 20% of compounds out, as long as all compounds within recover 65-135%	1) Perform maintenance as needed, recalibrate. 2) Note outliers in narrative.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8270* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Continuing Calibration Std (CCAL)	Laboratory Analytical Accuracy	1) Every 12 hrs prior to analysis of samples 2) Concentration level near midpoint of curve 3) Must contain all target analytes 4) Full Scan: Percent difference or percent drift (%D) must be ≤20 for CCCs and should be ≤30 for all other compounds. SIM: Percent difference or percent drift (%D) must be should be ≤30 for all compounds	NO	Recalibrate as required by method (1) if %D of any CCC >20 or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in case narrative.
Method Blanks	Laboratory Contamination Evaluation	1) Extracted every 20 or every batch, whichever is more frequent. 2) Matrix specific 3) Target analytes must be <RL except for common lab contaminants which must be <3x the RL (Contaminants are phthalates)	YES	Locate source of contamination and correct problem. Reanalyze method blank.	1) Report non-conformances in case narrative. 2) All results for compounds present in method blank must be "B" flagged if detected in samples associated with the method blank. 3) If re-extraction performed within holding time, report only compliant data. If re-extraction performed outside holding time report all data.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8270* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	1) Every 20 samples or each batch, whichever is more frequent. 2) Concentration level must be near or at the mid-point of the initial calibration. 3) Must contain all target analytes 4) Matrix and preservative specific 5) Laboratory determined percent recovery limits must be between 40-140% for base-neutrals and 30-130% for acid compounds. 6) Laboratories may spike blank soil or water for LCS	YES	Recalculate the percent recoveries Reanalyze the LCS Re-extract LCS and samples if >20% compounds outside acceptance criteria Locate & correct problem, reanalyze associated samples	1) Report non-conformances in case narrative. 2) Individual laboratories must identify and document problem analytes which routinely fall outside the limits. Any exceedances must be noted in narrative. Data to support laboratory problem compounds kept on file at lab for review during audit 3) If re-extraction performed within holding time, report only compliant data. If re-extraction performed outside holding time report all data.
Site Specific Matrix Spike/Matrix Spike Duplicate	Precision and Accuracy in Sample Matrix	1) Every 20 samples per matrix 2) Spike concentration in lower part of calibration curve. 3) Must contain all target analytes 4) Laboratory determined percent recovery limits must be between 40-140% for base-neutrals and 30-130% for acid compounds. 5) RPD's ≤ 20% for waters and ≤ 30% for soils.	YES (If analyzed)	If compounds out compare to LCS; if LCS recoveries in note in narrative; if LCS compounds out note in narrative probable lab error	Note outliers in narrative

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8270* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards	Laboratory and Method Accuracy in Sample Matrix	1) Full Scan Minimum of six IS's across GC run. SIM: Number of IS's will vary depending on number of analytes of interest. IS's must elute reasonably close to analytes and of similar class. 2) Area counts –50 to +100% of areas in associated continuing cal check. 3) Retention times of IS's ±30 seconds of associated continuing cal check.	NO	If any IS outside criteria, reanalyze sample extract.	1) Note exceedances in narrative. 2) If reanalysis confirms matrix interference report all results. 3) If reanalysis does not confirm matrix interference, report only compliant data. 4) If reanalysis outside holding time, report both sets of data.
Surrogates	Accuracy in Sample Matrix	1) Minimum 3 base-neutral and 3 acid surrogates across retention times of GC run. See Table 2B for recommended compounds. 2) Soil recovery limits lab generated and within 30-130%. 3) Water recovery limits lab generated and within 30-130% for base-neutrals, 15-110% for acidic compounds.	YES	Allowed one acid or one base-neutral surrogate out as long as above 10% rec. If any one surrogate <10% rec or if any two in a fraction out, re-extract. If surrogate diluted out below lowest calibration std, no recovery criteria.	1) Note exceedances in rrative. 2) If re-extraction confirms matrix interference or if re-extraction outside holding times report all results. 3) If re-extraction results in criteria and in holding time, report only compliant data.
Quantitation	N/A	1) Quantitation must be based on IS method. 2) Laboratory must use average RF or linear regression from initial calibration. 3) IS used for quantitation closest eluting to analyte.	N/A	N/A	If the average RF or linear regression not used for quantitation (e.g. quadratic equation) lab must note in narrative with list of affected analytes. Quadratic or polynomial fits require 6 & 7 calibration points.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8270* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	N/A	1) The laboratory should report only concentrations detected above the sample specific RL. 2) Concentrations below the reporting limit (RL) should be reported as "ND" with the sample specific RL also reported 3) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for both sets of data. Compounds which exceed the linear range should be flagged ("E" flag). Do not report more than two sets of data per sample. 4) If a dilution is performed, the highest detected analyte must be in the upper 60% of the calibration curve, unless there are non-target analytes whose concentrations are so high as to cause damage to the instrumentation or saturate the mass spectrometer 5) Refer to Appendix A for guidance on reporting TIC's	N/A	N/A	1) Qualification of results reported below the RL is required. 2) Performance of dilutions must be documented in the case narrative 3) TIC's will be evaluated according to Appendix A.

Notes for Table 1A:

* Refers to latest published version of SW-846 Method 8270.
GC/MS = Gas Chromatography/Mass Spectrometry
DFTPP = Decafluorotriphenylphosphine
%RSD = Relative Percent Standard Deviation
EP = Environmental Professional
r = Correlation Coefficient
RPD = Relative Percent Difference
CCC = Calibration Check Compound
N/A = Not Applicable

Note 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.Potentially Difficult Compounds include dimethyl phthalate, 4-notrophenol, phenol, 4-methylphenol, 2-methylphenol, 2,4-dinotrophenol, pentachlorophenol, and 4-chloroaniline.

1.5 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 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 supplies.

1.5.1 Reporting of Tentatively Identified Compounds (TICs)

If evaluated, all TICs that meet the chromatographic criteria presented in Appendix A of this method must be reported by the laboratory either in the Environmental Laboratory Report or in the Environmental Laboratory's case narrative. In turn, the EP must include a discussion regarding the disposition of all reported TICs as part of the RSR submission to DEP. 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 in order to meet analytical holding times.

1.6 Analyte List for SW-846 Method 8270

The Connecticut DEP 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 Remediation Standard Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

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. DEP 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.

The Reporting Limit (RL) is based upon the lowest standard in the initial calibration. . It is the responsibility of the EP to specify to the laboratory the detection limits required for the samples. In order to meet the limits 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 narrative.

1.7 Routine Reporting Deliverables for Method 8270

The following table (Table 1.2) 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.

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 Reporting Limit). The reporting limit for each compound in each sample must be listed on the report and take into account the exact sample mass, any dilution factors, percent moisture, etc.

Compounds detected above the reporting limit in blanks and found in samples, also above the reporting limit, 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.

Table 1.2 Report Deliverables

PARAMETER	DELIVERABLE	COMMENTS
GC/MS Tunes	NO	Note non-conformances in narrative
Initial Calibration	NO	Note non-conformances in narrative
Continuing Calibration	NO	Note non-conformances in narrative
Method Blanks	YES	Note non-conformances in narrative. Flag all positive results above RL with "B" flag.
Lab Control Sample (LCS)	YES	Note non-conformances in narrative
Site Specific Matrix Spike/ Matrix Spike Duplicate	YES (If requested)	Note non-conformances in narrative
Surrogate Recoveries	YES	Note non-conformances in narrative
Internal Standard Areas	NO	Note non-conformances in narrative
Tentatively Identified Compounds	YES (If requested)	Flag all values as estimated ("J" Flag)
General Reporting Issues	YES	Note non-conformances in narrative
QA/QC Certification Form	YES	Signed by laboratory director or his/her designee.

Table 1B Analyte List For SW-846 Method 8270

ANALYTE	CAS NUMBER	NOTES
Acenaphthene	83329	
Acenaphthylene	208968	
Aniline	62533	
Anthracene	120127	
Benzo(a)anthracene	56553	
Benzo(b)fluoranthene	205992	
Benzo(g,h,i)perylene	191242	
Benzo(k)fluoranthene	207089	
Benzo(a)pyrene	50328	
Bis(2-chloroethyl)ether	111444	
Bis(2-chloroethoxy)methane	111911	
Bis(2-chloroisopropyl)ether	108601	See 1
Bis(2-ethylhexyl)phthalate	117817	
4-Bromophenyl-phenylether	101553	
Butylbenzylphthalate	85687	
Carbazole	86748	
4-Chloroaniline	106478	
4-Chloro-3-methylphenol	59507	
2-Chloronaphthalene	91587	
2-Chlorophenol	95578	
4-Chlorophenyl-phenylether	7005723	
Chrysene	218019	
Dibenzofuran	132649	
Dibenzo(a,h)anthracene	53703	
3,3'-Dichlorobenzidine	91941	
2,4-Dichlorophenol	120832	
Diethylphthalate	84662	
2,4-Dimethylphenol	105679	
Dimethylphthalate	131113	
Di-n-butylphthalate	84742	
4,6-Dinitro-2-methylphenol	534521	
2,4-Dinitrophenol	51285	
2,4-Dinitrotoluene	121142	
2,6-Dinitrotoluene	606202	
Di-n-octylphthalate	117840	
Fluoranthene	206440	
Fluorene	86737	
Hexachlorobenzene	118741	

ANALYTE	CAS NUMBER	NOTES
Hexachlorobutadiene	87683	
Hexachlorocyclopentadiene	77474	
Hexachloroethane	67721	
Indeno(1,2,3-cd)pyrene	193395	
Isophorone	78591	
2-Methylnaphthalene	91576	
2-Methylphenol (o-Cresol)	95487	
4-Methylphenol (p-Cresol)	106445	See 2
Naphthalene	91203	
2-Nitroaniline	88744	
3-Nitroaniline	99092	
4-Nitroaniline	100016	
Nitrobenzene	98953	
2-Nitrophenol	88755	
4-Nitrophenol	100027	
N-nitrosodiphenylamine	86306	
N-Nitroso-di-n-propylamine	621647	
Pentachloronitrobenzene	82688	
Pentachlorophenol	87865	
Phenanthrene	85018	
Phenol	108952	
Pyrene	129000	
Pyridine	110861	
1,2,4,5-Tetrachlorobenzene	95943	
1,2,4-Trichlorobenzene	120821	
2,4,5-Trichlorophenol	95954	
2,4,6-Trichlorophenol	88062	

Notes:

1. Also known as 2,2'oxybis(1-chloropropane).
2. 3-Methylphenol and 4-Methylphenol cannot be separated chromatographically, and are calibrated and reported as 4-Methylphenol

Table 1C GC/MS Tune Criteria for DFTPP

m/z	Required Intensity (relative abundance)
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	<1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

The mass spectrum of DFTPP should be acquired in the following manner. Three scans (the peak apex and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and may be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not subtract part of the DFTPP peak. Alternative DFTPP criteria, such as the Method 525 or CLP criteria, can be utilized provided all samples, standards, blanks, etc. are analyzed using the same GC/MS tuning criteria is. If alternative approaches are utilized, the approach must be documented in the laboratory standard operating procedure. The laboratory is not allowed to vary its approach from day to day in order to in order to pass a tune on an instrument requiring maintenance.

Table 2A Sample Containers, Preservation and Holding Times

MATRIX	CONTAINER	PRESERVATIVE	HOLDING TIME
Aqueous with no chlorine present	1-liter amber glass bottle with Teflon line cap	Store at 4 ± 2º C.	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.	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. (Note 1)
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.

Notes:

The number of sample containers is optional. Laboratories should supply enough containers to allow for any reanalysis or breakage.

Note 1: 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.

Table 2B Recommended Internal Standards and Surrogates

Compound Type	Name	Comment
IS	Acenaphthene-d10	
IS	Chrysene-d12	
IS	1,4-Dichlorobenzene-d4	
IS	Naphthalene-d8	
IS	Perylene-d12	
IS	Phenanthrene-d10	
SURR	2-Fluorobiphenyl	BN
SURR	Nitrobenzene-d5	BN
SURR	Terphenyl-d14	BN
SURR	2-Fluorophenol	Acid
SURR	Phenol-d6	Acid
SURR	2,4,6-Tribromophenol	Acid
SURR	Fluoranthene-d10	Alternative surr for SIM
SURR	Benzo(a)pyrene-d12	Alternative surr for SIM

Appendix A

Laboratory Requirements For Evaluation of

Tentatively Identified Compounds

Method 8270

A-1. Chromatographic Criteria

A-1.1 Initially include all of the non-target compounds that elute after the first internal standard and within 3 minutes of the last target analyte. The peak area count of the unknown compound must also be ≥ 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 case 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 case 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. This only applies to peaks having an area >10% of the nearest internal standard.

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 case narrative or by using a "J" flag designation.

NOTE: In most circumstances the laboratory must order standards in order 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.