

State of Connecticut  
Department of Energy and Environmental Protection  
Recommended Reasonable Confidence Protocols  
Quality Assurance and Quality Control Requirements  
Volatile Organics by Method 8260, SW-846  
Version 4.0  
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Written by the Connecticut DEEP QA/QC Workgroup

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## Acronym List

<b><u>ACRONYM</u></b>	<b><u>DEFINITION</u></b>
BFB	Bromofluorobenzene
BTEX	Benzene, toluene, ethylbenzene, xylenes
CASN	Chemical Abstracts Service Number
CCV	Continuing calibration verification
%D	Percent difference or percent drift
DCB	Dichlorobenzene
DEEP	CT Department of Energy and Environmental Protection
DF	Dilution factor
EDB	Ethylene dibromide
EP	Environmental Professional
ETBE	Ethyl tertiary butyl ether
g	grams
GC	Gas chromatograph
GC/MS	Gas chromatography/mass spectrometry
HCl	Hydrochloric acid
ICV	Initial calibration verification
LCS	Laboratory control sample
LLOQ	Lower Limit of Quantitation
MEK	Methyl ethyl ketone
MIBK	Methyl isobutyl ketone
mL	Milliliters
MS	Matrix spike
MSD	Matrix spike duplicate
MTBE	Methyl tertiary butyl ether
NA	Not applicable
NaHSO <sub>4</sub>	Sodium bisulfate
OHM	Oil and Hazardous Materials
QA	Quality assurance
QC	Quality control
r/r <sup>2</sup>	Correlation coefficient
%R	Percent recovery
%RSD	Percent relative standard deviation
RCP	Reasonable Confidence Protocol
RF	Response factor
RL	Reporting limit
RPD	Relative percent difference
RSR/RSRs	Remediation Standard Regulations
SIM	Selective ion monitoring
TAME	Tertiary amyl methyl ether
TCE	Trichloroethene
THF	Tetrahydrofuran
TICs	Tentatively identified compounds
TSP	Trisodium phosphate dodecahydrate
UCM	Unresolved complex mixture
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µL	microliters
VOCs	Volatile organic compounds
VPH	Volatile petroleum hydrocarbons

## **1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 8260**

### **1.1 Method Overview**

SW-846 Method 8260 is a purge and trap gas chromatography/mass spectrometry (“GC/MS”) procedure used for the analysis of volatile organic compounds (“VOCs”) 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 the purge and trap system as described in SW-846 Methods 5030 and 5035.

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

### **1.2 Summary of SW-846 Method 8260**

The VOCs are introduced into the gas chromatograph (“GC”) by a purge and trap device or other technique. The analytes are then introduced directly to a capillary column by ballistic heating or cryo-focused onto a capillary pre-column before being flash evaporated to 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.

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 minimum five-point calibration curve.

### **1.3 Method Interferences**

Refer to SW-846 Method 8260 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 SW-846 Method 8260 include, but are not limited to, volatiles chemicals (solvents) in the laboratory, impurities in the purge gas, sorbent trap break down products or impurities, 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 concentrations 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 VOCs. Autosampler positions on the purge and trap unit 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 method blanks. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run.

Many analytes exhibit low purging efficiencies from a 25 mL sample. This often results in significant amounts of these analytes remaining in the sparging flask. Refer to the section on interferences of SW-846 Method 8260 for detailed approaches to minimizing these interferences, as well as other special precautions associated with methylene chloride, a common laboratory contaminant.

### 1.3.3 Other Potential Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank carried through sampling and subsequent storage and handling can serve as a check on such contamination. Laboratories should have a storage blank program as part of their QA/QC plan to monitor refrigerators for potential cross contamination.

The use of sodium bisulfate as the low-level preservation method for solid samples with high organic matter or humic material content has been known to result in the formation of acetone and methyl ethyl ketone (“MEK” or 2-butanone) at potentially significant concentrations in samples. Sodium bisulfate preservation must **never** be used when these conditions are present or suspected. It should be noted that freezing (<-7°C or 20°F) and not sodium bisulfate addition, is the preferred low-level preservation method for solid samples (See Table 4.0).

Use of methanol in the high level solid-preservation method may result in the detection of MEK at trace levels in samples due to the presence of MEK as a methanol contaminant.

The inherent limitation of methanol preservation with respect to the evaluation of matrix spike recoveries is more than compensated for by the marked improvement in sample integrity and conservation/recoveries of the volatile analytes of concern from soil matrices by minimizing volatilization losses.

## 1.4 Alternative Sample Introduction Methods

Various alternatives are provided in SW-846 Method 8260, for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Quality control procedures to ensure proper operation of the various sample introduction techniques may be found in SW-846 Methods 3500 and 5000, respectively.

This guidance document is primarily intended to provide QA/QC requirements and performance standards for SW-846 Method 8260 using conventional purge and trap sample introduction via Methods 5030 (ambient temperature) and 5035 for aqueous and solid samples, respectively. If other sample introduction methods are required and utilized because of analytical circumstances, the laboratory must provide a full explanation and justification in the laboratory report narrative. This narrative should also include details and results of the QC samples and calibrations associated with the different sample introduction method.

## 1.5 Quality Control Requirements for SW-846 Method 8260

### 1.5.1 Reporting Limits/Lower Limits of Quantitation for SW-846 Method 8260

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, sample introduction method, and moisture content. Table 1.0 lists approximate RL/LLOQs for various matrices utilizing the standard quadrupole mass spectrometer. Solid matrices in this table assume 100% solids.

**Table 1.0: Typical Reporting Limits / Lower Limit of Quantitation<sup>1</sup>**

Matrix	Typical Reporting Limit
Water	0.5 - 5.0 µg/L
Soil, Low Level	5 µg/Kg
Soil, High Level (methanol preserved)	100 - 500 µg/Kg
<sup>1</sup> 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 (“SIM”), an ion trap mass spectrometer, or newer instrumentation. Certain analytes, notably water-soluble compounds such as acetone, 2-hexanone, etc., have poor purging efficiencies. This will mandate higher calibration levels for these type compounds and therefore higher RLs/LLOQs. Some analytes may require heated purge and trap to achieve the required RL/LLOQ. Therefore, oxygenates and other compounds susceptible to acid hydrolysis should not be preserved with acid if heated purge and trap is to be used.

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.

### 1.5.2 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 volatile organics.

Refer to SW-846 Method 8000 for general QC requirements for all chromatographic methods, including SW-846 Method 8260. These requirements ensure that each laboratory maintain a formal 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 8260.

The minimum requirements for a formal QA program include Initial Demonstration of Laboratory 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”) and matrix spike duplicates (“MSD”) to assess precision. The use of site-specific MS/MSD’s 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/MSD’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 1.5 and Table 1A of this RCP and presented in SW-846 Method 8000. The IDOC must include the following elements provided in Table 2.0:

**Table 2.0: IDOC Requirements**

<b>QC Element</b>	<b>Performance Criteria</b>
4-Bromofluorobenzene (BFB) Tuning	See Method 8260
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, MS/MSD 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.

### 1.5.3 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260

Specific QA/QC requirements and performance standards for SW-846 Method 8260 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. 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 SW-846 Method 8260**

<b>Required QC Parameter</b>	<b>Data Quality Objective</b>	<b>Required Performance Standard</b>	<b>Required Deliverable</b>	<b>Required Corrective Action</b>	<b>Required Analytical Response Action</b>
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 1.5.2 of this RCP.	NA
GC/MS Tunes with BFB	Inter-laboratory Consistency & Comparability	(1) Criteria listed in SW-846 8260 (the same criteria must be used for all analyses). (2) Required prior to ICAL. NOTE: It is strongly recommended the analyst monitor BFB periodically and perform maintenance as necessary.	No	Perform instrument maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified.



Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration ("ICAL")	Laboratory Analytical Accuracy	<p>(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed (e.g., if the system is retuned).</p> <p>(2) Minimum of 5 standards (or 6 if non-linear regression used).</p> <p>(3) Low standard must be <math>\leq</math>RL/LLOQ.</p> <p>(4) %RSD &lt;20, <math>r &gt; 0.99</math> (linear regression), or <math>r^2 &gt; 0.99</math> (non-linear regression) for each target analyte.</p> <p>(5) If %RSD &gt;20, linear or non-linear regression must be used.</p> <p>(6) Minimum RFs for each compound as per SW-846 8260 for lowest concentration standard and for average RF.</p> <p>(7) Must contain all target analytes.</p> <p>(8) Calibration must be performed under the same conditions as the samples (e.g., heated purge).</p> <p>(9) If autosampler used to spike surrogates in calibration standards, one-point calibration with 5 standards acceptable for surrogates.</p> <p>(10) 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%.</p> <p>(11) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates, and internal standards.</p> <p>(12) If regression is used, must not be forced through the origin unless allowed by SW-846.</p>	No	<p>(1) Recalibrate if &gt;10% of target analytes exceed %RSD, "r", or "r<sup>2</sup>" criteria.</p> <p>(2) If &lt;10% of compounds exceed criteria, recalibration is not required as long as %RSD &lt;40, <math>r &gt; 0.98</math>, or <math>r^2 &gt; 0.98</math>.</p> <p>(3) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either:</p> <p>(a) The RL/LLOQ must be reported as an estimated value<sup>1</sup>, or</p> <p>(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.</p>	<p>(1) Sample analysis cannot proceed without a valid initial calibration.</p> <p>(2) Report non-conforming compounds (%RSD &gt;20, <math>r &lt; 0.99</math>, <math>r^2 &lt; 0.99</math> or minimum RF not met) in laboratory report narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory report narrative along with the compounds affected.</p>

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
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 <sup>1</sup> which must exhibit percent recoveries between 40-160%.	No	(1) Compounds must recover within 70-130% (2) Laboratories are allowed to have 20% of compounds out, as long as all compounds within recover 40 -160%. (3) 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 <sup>1</sup> , report non-conforming compounds in laboratory report narrative.
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) %D must be ≤20 for each target analyte. (5) Minimum RFs as per SW-846 8260. (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% of target analytes 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 on-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Method Blank ("MB")	Laboratory Method Sensitivity & Contamination Evaluation	(1) Every ≤20 field samples, or every batch, whichever is more frequent prior to sample analysis and after calibration standards. (2) Matrix and preservative-specific (e.g., water, methanol). (3) Target analytes must be <RL/LLOQ except for common laboratory contaminants (acetone, methylene chloride, and MEK) which must be <5x the RL/LLOQ.	Yes	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; 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 reanalysis is not possible, report non-conformance in laboratory report narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory report narrative. (3) If reanalysis is performed within holding time and yields acceptable method blank results, only report results of the reanalysis. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample ("LCS")	Laboratory Analytical Accuracy	<p>(1) Every <math>\leq 20</math> field samples or for each new tune clock, whichever is more frequent.            (2) Concentration level near midpoint of curve.            (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol).            (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes<sup>1</sup> which must exhibit percent recoveries between 40-160%.            (6) Can also be used as CCV.</p> <p>NOTE: If used as continuing calibration standard, must be evaluated using Performance Standards, Corrective Actions, and Analytical Response Actions listed above for Continuing Calibration.</p> <p>(7) Lab may have up to 10% of compounds out of criteria as long as within 40-160% recovery.</p>	Yes	<p>(1) Locate source of problem; reanalyze LCS and associated samples if &gt;10% of all analytes are outside of criteria.            (2) If &lt;10% of compounds are outside of the acceptance criteria, reanalysis is not required as long as recoveries are &gt;40%.            (3) If &gt;10% of compounds are above the acceptance criteria (&gt;130%), reanalysis is not required if affected compounds were not detected in associated samples.</p>	<p>(1) If sample reanalysis is not possible, report non-conformance in laboratory report narrative.            (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes<sup>1</sup>, report non-conforming compounds in laboratory report narrative.            (3) If re-analysis is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-analysis only.            (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.</p>

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
LCS Duplicate ("LCSD")	Laboratory Analytical Accuracy & Precision	(1) Every ≤20 field samples or for each new tune clock, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix and preservative-specific (e.g., water, methanol). (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes <sup>1</sup> which must exhibit percent recoveries between 40-160%. (6) Recommended to be run immediately after LCS in analytical sequence. (7) RPDs must be <20% for waters and solid.	Yes	(1) Locate source of problem; reanalyze LCS and associated samples if >10% of all analytes are outside of the recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, reanalysis is not required as long as recoveries are >40%. (3) If >10% of compounds are above the recovery acceptance criteria (>130%), reanalysis is not required if affected compounds were not detected in associated samples.	(1) If sample reanalysis is not possible, report non-conformance in laboratory report narrative. (2) If recovery is outside of 70-130% for any analyte, including "difficult" analytes <sup>1</sup> or RPD >20 for any analyte, including "difficult" analytes <sup>1</sup> , report non-conforming compounds in laboratory report narrative. (3) If re-analysis is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the laboratory must report results of both the initial analysis and re-analysis.
Matrix Spike/ Matrix spike Duplicate ("MS/MSD")  (Site-specific)	Method Accuracy & Precision 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 must be between 70-130% for target analytes except for "difficult" analytes <sup>1</sup> which must exhibit percent recoveries between 40-160%. (6) RPDs <20% for waters and <30% for solids. (7) Field blanks, trip blanks, etc. cannot be used for MS/MSDs.	Yes  ONLY when requested by the 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 surrogates, at retention times across GC run. (2) Percent recoveries must be between 70-130% for individual surrogate compounds.	Yes	<p>If one or more surrogates are outside of limits, reanalyze sample unless one of the following exceptions applies:            (1) Obvious interference present (e.g., UCM).</p> <p>NOTE: If obvious interference is present and surrogate recovery would cause rejection of data (i.e., &lt;10%), reanalyze sample on dilution.            (2) Methanol-preserved samples: re-analysis is not required if % moisture &gt;25 and surrogate recovery is &gt;10%.            (3) If one or more surrogates exhibit high recoveries and target analytes are not detected in sample, reanalysis is not required.</p>	<p>(1) Report non-conformances in laboratory report narrative.            (2) If reanalysis yields similar surrogate non-conformances, the laboratory must report results of both analyses.            (3) If reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the reanalysis only.            (4) If reanalysis is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses.            (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.</p>

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 internal standards at retention times across GC run. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated CCV. (3) Retention times of internal standards must be within $\pm 30$ seconds of retention times in associated CCV.	No	If one or more internal standards are outside of limits, reanalyze sample unless obvious interference present (e.g., UCM).  NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), re-analyze sample on dilution.	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 reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If re-analysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Do not report concentrations below the RL/LLOQ.	NA	NA	If the average RF or linear regression was not used for analyte quantitation (e.g., quadratic equation), it must be noted in the laboratory report narrative along with a list of affected analytes.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Analyte Identification	NA	Refer to SW-846 8260.	NA	NA	NA
General Reporting Issues	NA	<p>(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.</p> <p>(2) Concentrations below the RL/LLOQ are reported as “ND” with the sample-specific RL/LLOQ also reported.</p> <p>(3) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported.</p> <p>(3) Refer to Section 1.10 and Appendix A of this RCP, for guidance on TICs</p> <p>(4) All soil/sediment sample results preserved in methanol must be corrected for the methanol dilution as per Section 1.8.2 of this RCP.</p> <p>(5) Results for soils/sediments must be reported on a dry-weight basis for comparison to RSR regulatory standards.</p>	NA	NA	<p>(1) Qualification of the data is required if reporting values below the sample-specific RL/LLOQ.</p> <p>(2) Complete analytical documentation for diluted and undiluted analyses must be documented in laboratory report narrative and be maintained in laboratory records.</p> <p>(3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix A of this RCP.</p> <p>(4) The performance of dilutions must be documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory report narrative.</p> <p>(5) If samples are not properly preserved (pH &gt;2 for aqueous samples, solid samples not completely covered with appropriate preservative) or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report narrative.</p> <p>(6) If samples are preserved and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative.</p>



Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
<p><sup>1</sup>Potentially Difficult Compounds include: acetone, bromoform, bromomethane, 2-butanone (MEK), chloroethane, chloromethane, 1,2-Dibromo-3-chloropropane, dibromochloromethane, dichlorodifluoromethane, cis-1,3-Dichloropropene, 1-4, dioxane, 2-hexanone, hexachlorobutadiene, 4-methyl-2-pentanone (MIBK), naphthalene, styrene, 1,1,2,2-Tetrachloroethane, and trichlorofluoromethane.</p> <p>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.</p>					

## **1.6 Special Analytical Considerations for SW-846 Method 8260**

The following section highlights potential issues that may be encountered with the analysis of VOCs using this protocol.

Analytes with poor purging efficiency at ambient temperature, may require the heated purge-and-trap option if lower RL/LLOQs are required.

Aqueous samples submitted for analysis of oxygenates, and other compounds susceptible to hydrolysis should not be preserved with acid if heated purge-and-trap (>40°C) is used as the sample introduction method. See Table 4.0 for the preferred preservation technique under this condition.

Under certain conditions, select VOCs may be potentially reactive (i.e., unstable and susceptible to acid hydrolysis, abiotic degradation and/or loss during storage).

The recovery of matrix spikes from a soil/sediment sample that has been preserved with methanol cannot be used to directly evaluate matrix-related bias/accuracy in the conventional definition of these terms. QC parameters expressed in terms of these percent recoveries (“%R”) may be more indicative of the variabilities associated with the analytical system (sample processing, introduction, and/or component separation). This inherent limitation of methanol preservation with respect to the evaluation of matrix spike recoveries is more than compensated for by the marked improvement in sample integrity and conservation/recoveries of the volatile analytes of concern from soil/sediment matrices by minimizing volatilization losses.

1,4-Dioxane is included on the analyte list provided in Table 1B. The analytical sensitivity (i.e., RL/LLOQ) for this compound (200 – 500 µg/L in water) is not adequate to evaluate compliance with some RSR Additional Polluting Substances (“APS”) criteria if conventional (ambient temperature) purge-and-trap sample introduction is utilized. If 1,4-Dioxane is not a contaminant of concern for the site, conventional purge-and-trap sample introduction (ambient temperature) may be used for sample analysis.

- If 1,4-dioxane is a contaminant of concern for the site, special analytical techniques, as listed below, must be utilized to evaluate compliance with RSR criteria.
- Heated (80±5°C) purge-and-trap with SIM analysis by SW-846 Method 8260 is an acceptable approach for aqueous and solid samples. However, if elevated concentrations of other chlorinated VOCs are present in the sample, this approach may not be preferable due to the likely contamination/saturation of the trap during the analysis.
- Extraction using SW-846 methods 3510 or 3535 followed by isotope dilution analysis using SW-846 method 8270, is an acceptable approach for aqueous samples.

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.

### 1.7 Analyte List for SW-846 Method 8260

The DEEP analyte list for SW-846 Method 8260 is presented in Table 1B. The compounds listed are readily analyzable by SW-846 Method 8260. 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 8260**

Analyte	CASN	Analyte	CASN
Acetone	67641	cis-1,3-Dichloropropene	10061015
Acrylonitrile	107131	trans-1,3-Dichloropropene	10061026
Tert-Amyl Methyl Ether (TAME)	994058	Diethyl Ether 60297	60297
Benzene	71432	1,4-Dioxane <sup>1</sup>	123911
Bromobenzene	108861	Ethylbenzene	100414
Bromochloromethane	74975	Ethyl Tertiary Butyl Ether (ETBE)	637923
n-Butylbenzene	104518	Hexachlorobutadiene	87683
Sec-Butylbenzene	135988	2-Hexanone	591786
Tert-Butylbenzene	98066	Isopropylbenzene (Cumene)	98828
Bromodichloromethane	75274	4-Isopropyltoluene	99876
Bromoform	75252	Methylene Chloride	75092
Bromomethane	74839	4-Methyl-2-pentanone (MIBK)	108101
2-Butanone (MEK)	78933	Methyl-tert-butyl ether (MTBE)	1634044
Carbon Disulfide	75150	Naphthalene	91203
Carbon Tetrachloride	56235	n-Propylbenzene	103651
Chlorobenzene	108907	Styrene	100425
Chloroethane	75003	1,1,1,2-Tetrachloroethane	630206
Chloroform	67663	1,1,2,2-Tetrachloroethane	79345
Chloromethane	74873	Tetrachloroethene (Perc)	127184
2-Chlorotoluene	95498	Tetrahydrofuran (THF)	109999
4-Chlorotoluene	106434	Toluene	108883
Dibromochloromethane	124481	1,2,3-Trichlorobenzene	87616
1,2-Dibromo-3-chloropropane (DBCP) <sup>1</sup>	96128	1,2,4-Trichlorobenzene	120821
1,2-Dibromoethane (EDB) <sup>1</sup>	106934	1,1,1-Trichloroethane	71556
Dibromomethane	74953	1,1,2-Trichloroethane	79005
1,2-Dichlorobenzene	95501	Trichloroethene (TCE)	79016
1,3-Dichlorobenzene	541731	Trichlorofluoromethane	75694
1,4-Dichlorobenzene	106467	1,2,3-Trichloropropane	96184
trans-1,4-Dichloro-2-butene	110576	Trichlorotrifluoroethane (Freon-113)	76131
Dichlorodifluoromethane	75718	1,2,4-Trimethylbenzene	95636
1,1-Dichloroethane	75343	1,3,5-Trimethylbenzene	108678
1,2-Dichloroethane	107062	Vinyl Chloride	75014
1,1-Dichloroethene	75354	o-Xylene <sup>2</sup>	95476
cis-1,2-Dichloroethene	156592	m-Xylene <sup>2</sup>	108383
trans-1,2-Dichloroethene	156605	p-Xylene <sup>2</sup>	106423
1,2-Dichloropropane	78875		
1,3-Dichloropropane	142289		
2,2-Dichloropropane	594207		
1,1-Dichloropropene	563586		

<sup>1</sup> These compounds require analysis by an alternative method to achieve applicable RSR criteria.  
<sup>2</sup> May be reported as total xylenes or any combination of the three isomers.

### 1.7.1 Additional Reporting Requirements for SW-846 Method 8260

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.

### 1.8 Routine Reporting Deliverables for SW-846 Method 8260

The following table (Table 3.0) 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 3.0: 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 Verification	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive results above RL/LLOQ 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 concentrations 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.

### 1.8.1 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, based upon the lowest calibration standard, the exact sample mass, any dilution factors, percent moisture, etc.
- Compounds detected above the RL/LLOQ in blanks and in samples 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.

### 1.8.2 Special requirements for methanol preserved soil/sediment samples

VOCs results for methanol preserved soil/sediment samples must be corrected for the Methanol Preservation Dilution Effect as discussed in SW-846 Method 8000. For methanol preserved samples the total methanol/water volume,  $V_t$ , is given by the following equation:

$$V_t = (\text{mL methanol}) + (\text{decimal \% moisture} \times \text{g sample})$$

This  $V_t$  value should be substituted into the equation for  $V_t$  in the equation presented in Method 8000.

### 1.9 Sample Containers, Preservations, and Holding Times

Table 4.0 identifies the type of containers, preservation requirements, and holding times dependent upon analyte and matrix.

**Table 4.0: Sample Containers, Preservations and Holding Times**

Matrix	Analyte	Container <sup>1</sup>	Preservative <sup>2,3</sup>	Holding Time <sup>4</sup>
Aqueous w/ no Residual Chlorine	VOCs	40mL VOCs vials w/ Teflon-lined septa screw caps and protect from light	Adjust pH to <2 by addition of HCl or NaHSO <sub>4</sub> to container before sampling. Cool to 4 ± 2°C, but not frozen. <sup>5</sup>	14 days
	MTBE & other fuel oxygenates with heated purge-and-trap (>40°C) sample introduction only.	40mL VOCs vials w/ Teflon-lined septa screw caps and protect from light.	0.7g of trisodium phosphate dodecahydrate (“TSP”) per 40 mL. Verify pH >11.0. Cool to 4 ± 2°C, but not frozen. <sup>6</sup>	14 days
	Volatile organics susceptible to acid hydrolysis, abiotic degradation or loss during storage.	40mL VOCs vials w/ Teflon-lined septa screw caps and protect from light.	Cool to 4 ± 2°C, but not frozen.	14 days
Aqueous with Residual Chlorine	Presence of chlorine residual is usually associated with drinking water samples. Collect sample in at least two 40mL VOCs vials w/ Teflon-lined septa screw caps containing either 35 mg of Ascorbic Acid or 3 mg of Sodium thiosulfate. If residual chlorine >5 mg/L, additional dechlorination agent may be required. After dechlorination is confirmed, preserve as above based on compound class.			
Soil and Sediment samples.	All VOCs with purge & trap ≤ 45°C. <sup>7</sup>	Samples should be collected and stored according to the DEEP <i>Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006.</i> <sup>8</sup>	Ice samples in field and proceed with preservation option selected. Preservation options include methanol, sodium bisulfate, and freezing. <sup>9</sup>	14 days if preserved. 48 hours if unpreserved. <sup>10</sup>

Matrix	Analyte	Container <sup>1</sup>	Preservative <sup>2,3</sup>	Holding Time <sup>4</sup>
High Conc. Waste Samples	All VOCs with purge & trap ≤ 45°C. <sup>7</sup>	Samples should be collected and stored according to the DEEP <i>Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006.</i> <sup>8</sup>	Ice samples in field and proceed with preservation option selected. Preservation options include methanol, sodium bisulfate, and freezing. <sup>9</sup>	14 days if preserved. 48 hours if unpreserved. <sup>10</sup>

<sup>1</sup>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.

<sup>2</sup>Preservation of samples by acidification to pH <2.0 and analysis within 14 days is considered a suitable preservation technique for samples not expected to contain reactive contaminants of concern.

<sup>3</sup>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 6°C are acceptable.

<sup>4</sup>If the holding time is exceeded by >2x the allowable holding time, data users should consider non-detect results as unusable and positive results as estimated with a significantly low bias.

<sup>5</sup>If samples effervesce upon addition of hydrochloric acid, samples must be collected unpreserved and stored at ≤ 6°C. Holding time is 7-days from collection.

<sup>6</sup>TSP may also be used to preserve samples for BTEX and/or VPH analysis (i.e., it would not be necessary to obtain samples in separate vials).

<sup>7</sup>If the purge temperature is >45°C, bisulfate cannot be used as degradation of certain analytes (e.g., MTBE) may occur.

<sup>8</sup>Laboratories are reminded to include a separate container for % solids determination.

<sup>9</sup>If samples effervesce upon addition of sodium bisulfate, then bisulfate cannot be used as a preservative. Another preservation option must be selected.

<sup>10</sup>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 48 hours.

\*\*EnCore Samplers may not be suitable for all soil types. See SW-846 Method 5035 and the DEEP *Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006* for guidance.

## **1.10 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 the 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 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.

### 1.10.1 Reporting of Tentatively Identified Compounds

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 require re-sampling to meet analytical holding times.



## **Appendix A: Laboratory Requirements for Evaluation of Tentatively Identified Compounds Method 8260**

### ***A-1. Chromatographic Criteria***

A-1.1 Initially include all the non-target compounds that have a peak area count  $\geq 10\%$  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 spectrometrists and the Organic Supervisor/Laboratory Director.

A-2.2 The spectral library match must be  $\geq 85\%$  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  $\pm 20\%$ .

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 After the above criteria are met, the top ten (10) compounds for VOCs, 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 number of peaks present, the laboratory must evaluate any peak where 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 internal standard 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.

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 to attain TIC results that are scientifically defensible.

## **Appendix B: Low Reporting Limit Modifications for Meeting Groundwater Protection Criteria**

### ***B-1. Introduction***

B-1.1 The Connecticut Remediation Standards require drinking water methods be used to analyze certain groundwater samples collected from a GA area. This modification to the DEEP Recommended Protocol for SW-846 Method 8260 may be used in place of Method 524.2 with the approval of the DEEP.

B-1.2 This modification should be used for clean samples. Samples with analytes present above the linear range of this method, without dilution, should be analyzed using the standard in the DEEP Recommended Protocols for SW-846 Method 8260. The upper linear range without dilution will vary with the laboratory, but is approximately 30 µg/L.

### ***B-2. QC Requirements***

B-2.1 All QC criteria specified in the DEEP Recommended Protocols for Method 8260 must be followed.

B-2.2 The instrument must be calibrated at or below the RL/LLOQ specified in Table B-1 of this appendix.

B-2.3 A 5 mL purge volume must be used for all samples and standards. Different purge volumes may be used as long as data quality objectives are met, including RL/LLOQ requirements.

B-2.4 A trip blank is required for all sampling events. The trip blank must accompany the sample containers from the laboratory to the site and back to the laboratory. If samples are collected on separate days, a trip blank is required for each day. Analysis of the trip blank is ONLY required if there are analytes detected in the associated samples.

B-2.5 A library search for TICs is required when using this method as specified in Appendix A of this document.

### ***B-3. Report Deliverables***

B-3.1 The laboratory shall report as specified in Table 3.0 of this RCP for Method 8260 and in Appendix A of this document.

**Table B-1: Reporting Limits for Method 8260 Low Reporting Limits/Lower Limits of Quantitation**

Analyte	CAS Number	Aqueous RL/LLOQ, µg/L	Analyte	CAS Number	Aqueous RL/LLOQ, µg/L
Acetone	67641	5.0	Hexachlorobutadiene	87683	0.45
Acrylonitrile	107131	0.5	2-Hexanone	591786	5.0
Benzene	71432	0.5	Isopropylbenzene (Cumene)	98828	0.5
Bromobenzene	108861	0.5	4-Isopropyltoluene	99876	0.5
n-Butylbenzene	104518	0.5	Methylene Chloride	75092	0.5
Sec-Butylbenzene	135988	0.5	4-Methyl-2-pentanone (MIBK)	108101	5.0
Tert-Butylbenzene	98066	0.5	Methyl-tert-butylether (MTBE)	1634044	0.5
Bromodichloromethane	75274	0.5	Naphthalene	91203	0.5
Bromoform	75252	0.5	n-Propylbenzene	103651	0.5
Bromomethane	74839	0.5	Styrene	100425	0.5
2-Butanone (MEK)	78933	5.0	1,1,1,2-Tetrachloroethane	630206	0.5
Carbon Disulfide	75150	0.5	1,1,2,2-Tetrachloroethane	79345	0.5
Carbon Tetrachloride	56235	0.5	Tetrachloroethene (Perc)	127184	0.5
Chlorobenzene	108907	0.5	Tetrahydrofuran (THF)	109999	5.0
Chloroethane	75003	0.5	Toluene	108883	0.5
Chloroform	67663	0.5	1,2,3-Trichlorobenzene	87616	0.5
Chloromethane	74873	0.5	1,2,4-Trichlorobenzene	120821	0.5
2-Chlorotoluene	95498	0.5	1,1,1-Trichloroethane	71556	0.5
4-Chlorotoluene	106434	0.5	1,1,2-Trichloroethane	79005	0.5
Dibromochloromethane	124481	0.5	Trichloroethene (TCE)	79016	0.5
1,2-Dibromo-3-chloropropane (DBCP) <sup>1</sup>	96128	Note 1	Trichlorofluoromethane	75694	0.5
1,2-Dibromoethane (EDB) <sup>1</sup>	106934	Note 1	1,2,3-Trichloropropane	96184	0.5
Dibromomethane	74953	0.5	Trichlorotrifluoroethane (Freon-113)	76131	0.5
1,2-Dichlorobenzene	95501	0.5	1,2,4-Trimethylbenzene	95636	0.5
1,3-Dichlorobenzene	541731	0.5	1,3,5-Trimethylbenzene	108678	0.5
1,4-Dichlorobenzene	106467	0.5	Vinyl Chloride	75014	0.5
trans-1,4-Dichloro-2-butene	110576	0.5	o-Xylene <sup>2</sup>	95476	0.5
Dichlorodifluoromethane	75718	0.5	m-Xylene <sup>2</sup>	108383	0.5
1,1-Dichloroethane	75343	0.5	p-Xylene <sup>2</sup>	106423	0.5
1,2-Dichloroethane	107062	0.5			
1,1-Dichloroethene	75354	0.5			
cis-1,2-Dichloroethene	156592	0.5			
trans-1,2-Dichloroethene	156605	0.5			
1,2-Dichloropropane	78875	0.5			
1,3-Dichloropropane	142289	0.5			
2,2-Dichloropropane	594207	0.5			
1,1-Dichloropropene	563586	0.5			
cis-1,3-Dichloropropene	10061015	0.5			
trans-1,3-Dichloropropene	10061026	0.5			
Ethylbenzene	100414	0.5			

<sup>1</sup>These compounds require analysis by either Methods 504.1 or other method approved by the Commissioner to achieve the RSR limit in aqueous samples.

<sup>2</sup>May be reported as total xylenes or any combination of the three isomers.