State Of Connecticut
Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Volatile Organics by Method 8260, SW-846

Version 3.0

July 2006

Written by the Connecticut DEP QA/QC Workgroup

Revision	Comments	Date
1.0	First version for publication	7/11/05
2.0	Corrected holding time for frozen samples	8/30/2005
3.0	Final version based upon public comments	July 2006

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1.0 QA/QC Requirements for Method 8260

1.1 Method Overview

Method 8260 is a purge and trap gas chromatography/mass spectrometry procedure used to determine volatile organic compounds (VOC'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. The sample introduction procedure requires the use of the purge and trap system as described in Methods 5030 and 5035. All method references are to the latest published version of the method found in Test Methods for Evaluating Solid Waste, SW-846.

1.1.1 Reporting Limits for Method 8260

The reporting limit (RL) for a compound is dependent on the concentration of the lowest standard in the initial calibration, sample weight/volume, sample introduction method, 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.

TABLE 1.0 Typical Reporting Limits

Matrix	Typical Reporting Limit
Water, 25 mL purge	0.50 ug/L
Water, 5 mL purge	5.0 ug/L
Soil, Low Level	5 ug/Kg
Soil, Medium Level	25 ug/Kg
Soil, High Level (methanol preserved)	500 ug/Kg

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 acetone, 2-hexanone, etc., have poor purging efficiencies. This will mandate higher calibration levels for these type compounds and therefore higher RL's. Some analytes may require heated purge and trap in order to achieve the required RL. 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, 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**
BFB 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 8260

The volatile compounds are introduced into the gas chromatograph 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 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. 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.

1.3 Method Interferences

1.3.1 Chemical Contaminants

Major contaminant sources for 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 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 VOC's. 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 at the same autosampler position as the high sample. 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 Section 3.0 of SW-846 Method 8260B 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 (<-7ºC or 20ºF) and not sodium bisulfate addition, is the preferred low-level preservation method for solid samples (See Table 2A).

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.

1.4 Alternative Sample Introduction Methods

Various alternatives are provided in SW-846 Method 8260B, Section 7.1, 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 8260B 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 Environmental Laboratory case 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 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 8260. 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 8260, Sections 8.0 and 9.0, respectively.

1.5.2 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 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 herein.

1.5.3 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 the 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 and should not be reported for this protocol. Trip blanks, field blanks, etc. should not be used for MS/MSD's.

1.5.4 Recovery of Matrix Spike (MS) and Matrix Spike Duplicate (MSD) with Methanol Preserved Soil/Sediment Samples

The recovery of matrix spikes from a soil/sediment that has been preserved with methanol cannot be used to directly evaluate matrix-related bias/accuracy in the conventional definition of these terms. Quality control 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). Because of this limitation, it is recommended that laboratories analyze standard reference materials and participate in relevant performance evaluation studies as frequently as possible. Recommended practices for additional quality assurance made be found in SW-846 Methods 5000 and 8000.

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.

1.5.5 Trip Blanks and Field Duplicates for SW-846 Method 8260 Analyses

Trip blanks and field blanks are highly recommended. The use of these type QC samples can be invaluable in determining if contamination occurred during shipping and /or collection of samples. In those instances where unexpected sample results are obtained, the results of which do not have trip blank and or field blank supporting data, could require sample recollection in order to verify the results.

1.5.6 GC/MS Tuning Criteria

The mass spectrometer must meet the tune criteria using 4-Bromofluorobenzene (BFB). The 8260 criteria are listed in table 1.3. If laboratories elect to use the 8260 criteria, 50 ng of BFB should be used. If laboratories utilize the Method 524.2 criteria, 25 ng of BFB must be used.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8260*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with BFB	Inter-laboratory consistency and comparability	1) Criteria listed in Table 4 of SW-846 Method 8260 (the same criteria must be used for all analyses) 2) Every 12 hours	NO	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration (ICAL)	Laboratory Analytical Accuracy	1) Minimum of 5 standards. (Note 1). Single point calibration allowed for surrogates. 2) Low standard must be ≤ reporting limit (RL) 3) % RSD ≤ 15 or "r" ≥ 0.990 for all compounds except CCC's, which must be ≤ 30% RSD or "r" ≥ 0.990 4) Must contain all target analytes 5) If regression is used, must not be forced through the origin. 6) 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.
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) Percent difference or percent drift (%D) must be ≤20 for CCCs and should be ≤30 for all other 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.

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8260* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Contamination Evaluation	1) Every 12-hrs shift or 20 samples, whichever is more frequent, prior to sample analysis and after calibration standards. 2) Matrix and preservative-specific (e.g. water, MeOH, NaHSO4) 3) Target analytes must be <RL except for common lab contaminants which must be <3x the RL (Contaminants are acetone, methylene chloride, and 2-butanone)	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.
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	1) Every 20 samples/matrix or for each new tune clock, 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 (e.g. water, MeOH, NaHSO4) 5) Laboratory determined percent recoveries must be between 70-130% for target compounds 6) Can also be used as CCAL 7) Lab may have up to 10% of compounds out of criteria as long as within 40-160% recovery. 8) Laboratories may spike blank soil or water for LCS (No 2nd source requirement)	YES	Recalculate the percent recoveries Reanalyze the LCS 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 70-130% limit. Any exceedances must be noted in narrative. Data to support laboratory problem compounds kept on file at lab for review during audit
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	1) Every 20 samples (Site specific MS/MSD's are strongly recommended 2) Matrix Specific, not required for trip blanks or field blanks 3) Must contain all target analytes 4) Laboratory determined percent recoveries should be between 70-130% for target compounds 5) RPD's should be ≤ 30% 6) Field blanks, trip blanks, etc. cannot be used for MS/MSD's.	YES (When requested)	Compare to LCS recoveries, narrate any non-conformances	Report non-conformances in case narrative

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8260* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Surrogates	Accuracy in Sample Matrix	1) A minimum of 3 surrogates must be added to all samples, blanks, etc prior to sample introduction 2) Evaluate recoveries in each sample 3) Laboratory determined percent recoveries must be between 70-130% for individual surrogate compounds. Laboratory determined recovery limits may be outside 70-130% limits for difficult matrices (e.g. waste, sludges, etc) 4) Single point calibration allowed allowed for surrogates. See Note 1	YES	If one or more surrogates are outside limits, reanalyze the sample unless one of the following exceptions occurs: 1) For methanol preserved samples reanalysis is not required if moisture >25% and recoveries are >10% 2) If one surrogate exhibits high recovery and associated target compounds are not detected in the sample, report data as is, no reanalysis required.	1) Note exceedances in narrative 2) If reanalysis confirms matrix interference, report both sets of results and note in narrative 3) If reanalysis performed in holding time and surrogate recoveries are in range, report only the compliant data 4) If reanalysis performed outside of holding time and surrogate recoveries are in range, report both sets of data, note in narrative
Internal Standards (IS)	Laboratory Analytical Accuracy and Method Accuracy in Sample	1) Laboratory must use a minimum of 3 IS at retention times across the GC run 2) Area counts in samples must be within –50% to +100% of the area counts in the associated CCAL 3) Retention times of IS must be within ± 30 seconds of the IS retention times in the associated CCAL	NO	If any IS is outside the QC limits, reanalyze the sample	1) Note exceedances in narrative 2) If reanalysis confirms matrix interference, report both sets of results and note in narrative 3) If reanalysis performed in holding time and IS are in criteria, report only the compliant data 4) If reanalysis performed outside of holding time and IS are in criteria, report both sets of data, note in narrative

Table 1A Specific QA/QC Requirements and Performance Standards for Method 8260* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Quantitation	N/A	1) Quantitation must be based on IS calibration 2) The laboratory must use the average RF or linear regression from the initial calibration for quantitation of each analyte 3) The IS used for quantitation must be the IS nearest to the retention time of the target analyte.	N/A	N/A	1) If the average RF or linear regression was not used for analyte quantitation (e.g. quadratic equation), it must be noted in the narrative along with a list of affected analytes 2) Laboratories must supply example calculations for those cases where the average RF or linear regression was not used
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) as "ND" with the reporting limit. 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 2 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 8260.
GC/MS = Gas Chromatography/Mass Spectrometry
BFB = 4-Bromofluorobenzene
%RSD = Relative Percent Standard Deviation
RF = Relative Response Factor
EP = Environmental Professional
r = Correlation Coefficient
RPD = Relative Percent Difference
CCC = Calibration Check Compound
N/A = Not Applicable

Potentially Difficult Compounds include acetone, bromomethane, chloroethane, dichlorodifluoromethane, dibromochloromethane, hexachlorobutadiene, 2-butanone (MEK), 4-methyl-2-pentanone, and trichlorofluoromethane.

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 $\geq$ 0.990. Surrogates may be calibrated using a single point, at the same concentration as added to all samples, blanks, etc.

1.6 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 potable* 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.

1.6.1 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 require re-sampling in order to meet analytical holding times.

1.7 Analyte List for SW-846 Method 8260

The Connecticut DEP (DEP) analyte list for SW-846 Method 8260 is presented in Table 1B. The compounds listed are readily determined by Method 8260 using conventional purge and trap GC/MS. Method SW-846 5030 should be used for aqueous samples and methanol extracts, SW-846 Method 5035 should be used for soil/solid samples to minimize the loss of volatile organics. Most of the compounds listed have Connecticut Remediation Standard Criteria or are listed in the Approved Criteria for Additional Polluting Substances. The remaining compounds were

selected based upon Drinking Water Monitoring requirements as described in Sections 19-13-B101 and 19-13-B102 of the Regulations of the Connecticut State Agencies.

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. 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 detection 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.

1.8 Routine Reporting Deliverables for Method 8260

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.

1.8.1 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 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.

1.8.2 Special requirements for methanol preserved soil/sediment samples

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

Vt = (mls methanol) + (decimal % moisture x g sample)

This Vt value should be substituted into the equation for Vt in Equation 11.10.2 of Method 8000C.

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 8260

ANALYTE	CAS NUMBER	NOTES
Acetone	67641	
Acrylonitrile	107131	
Benzene	71432	
Bromobenzene	108861	
n-Butylbenzene	104518	
Sec-Butylbenzene	135988	
Tert-Butylbenzene	98066	
Bromodichloromethane	75274	
Bromoform	75252	
Bromomethane	74839	
2-Butanone (MEK)	78933	
Carbon Disulfide	75150	
Carbon Tetrachloride	56235	
Chlorobenzene	108907	
Chloroethane	75003	
Chloroform	67663	
Chloromethane	74873	
2-Chlorotoluene	95498	
4-Chlorotoluene	106434	
Dibromochloromethane	124481	
1,2-Dibromo-3-chloropropane (DBCP)	96128	See 1
1,2-Dibromoethane (EDB)	106934	See 1
Dibromomethane	74953	
1,2-Dichlorobenzene	95501	
1,3-Dichlorobenzene	541731	
1,4-Dichlorobenzene	106467	
trans-1,4-Dichloro-2-butene	110576	
Dichlorodifluoromethane	75718	
1,1-Dichloroethane	75343	
1,2-Dichloroethane	107062	
1,1-Dichloroethene	75354	
cis-1,2-Dichloroethene	156592	
trans-1,2-Dichloroethene	156605	
1,2-Dichloropropane	78875	
1,3-Dichloropropane	142289	
2,2-Dichloropropane	594207	
1,1-Dichloropropene	563586	
cis-1,3-Dichloropropene	10061015	
trans-1,3-Dichloropropene	10061026	
Ethylbenzene	100414	

ANALYTE	CAS NUMBER	NOTES
Hexachlorobutadiene	87683	
2-Hexanone	591786	
Isopropylbenzene (Cumene)	98828	
4-Isopropyltoluene	99876	
Methylene Chloride	75092	
4-Methyl-2-pentanone (MIBK)	108101	
Methyl-tert-butylether (MTBE)	1634044	
Naphthalene	91203	
n-Propylbenzene	103651	
Styrene	100425	
1,1,1,2-Tetrachloroethane	630206	
1,1,2,2-Tetrachloroethane	79345	
Tetrachloroethene (Perc)	127184	
Tetrahydrofuran (THF)	109999	
Toluene	108883	
1,2,3-Trichlorobenzene	87616	
1,2,4-Trichlorobenzene	120821	
1,1,1-Trichloroethane	71556	
1,1,2-Trichloroethane	79005	
Trichloroethene (TCE)	79016	
Trichlorofluoromethane	75694	
1,2,3-Trichloropropane	96184	
Trichlorotrifluoroethane (Freon-113)	76131	
1,2,4-Trimethylbenzene	95636	
1,3,5-Trimethylbenzene	108678	
Vinyl Chloride	75014	
o-Xylene	95476	See 2
m-Xylene	108383	See 2
p-Xylene	106423	See 2

Notes:

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

2. May be reported as total xylenes or any combination of the three isomers.

Table 1.3 GC/MS Tune Criteria for BFB

m/z	Required Intensity (relative abundance)
50	15-40% of m/z 95
75	30-60% of m/z 95
95	Base peak, 100% relative abundance
96	5 – 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 – 9% of m/z 174
176	Greater than 95%, but less than 101% of m/z 174
177	5 – 9% of m/z 176

The mass spectrum of BFB 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 BFB. Do not subtract part of the BFB peak. Alternative BFB criteria, such as the Method 524.2 criteria, is allowed provided all samples, standards, blanks, etc. are analyzed using the same GC/MS tuning criteria. 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	ANALYTE	CONTAINER	PRESERVATIVE	HOLDING TIME
Aqueous with no chlorine present	All VOC's with purge & trap ≤ 45ºC.	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Adjust to pH < 2 with either HCl or sodium bisulfate at time of collection (Note 1). Store at 4 ± 2º C.	14 days
Aqueous with chlorine present	All VOC's with purge & trap ≤ 45ºC.	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Neutralize chlorine with either 25 mg ascorbic acid or 3 mg sodium thiosulfate. Adjust to pH < 2 with either HCl or sodium bisulfate (Note 1). Store at 4 ± 2º C.	14 days
Aqueous with no chlorine present	VOC's + MTBE with purge & trap >45ºC.	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Adjust to pH > 11 with 0.7 g trisodium phosphate at time of collection . Store at 4 ± 2º C.	14 days
Aqueous with chlorine present	VOC's + MTBE with purge & trap >45ºC.	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Neutralize chlorine with either 25 mg ascorbic acid or 3 mg sodium thiosulfate. Adjust to pH > 11 with 0.7 g trisodium phosphate. Store at 4 ± 2º C.	14 days

Notes:

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

Note 1: If samples effervesce upon addition of hydrochloric acid, samples must be collected unpreserved and stored at 4 ± 2º C. Holding time is 7-days from collection.

Table 2A Sample Containers, Preservation , and Holding Times

MATRIX	ANALYTE	CONTAINER	PRESERVATIVE	HOLDING TIME
Soil and Sediment samples.	All VOC's with purge & trap ≤ 45ºC. (Note 4)	Samples should be collected and stored according to DEP *Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006.* Laboratories are reminded to include a separate container for % solids determination.	Ice samples in field and proceed with preservation option selected. Preservation options include methanol, sodium bisulfate, and freezing. (See notes 2 & 3).	14 days if preserved. 48 hours if unpreserved. (Note 5).
High Conc. Waste Samples	All VOC's	Collect in screw top jar protected from light.	Cool 4 ± 2º C.	14 days

Notes:

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

Note 2: EnCore Samplers may not be suitable for all soil types. See Method 5035A in SW-846 and the DEP *Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006* for guidance.
Note 3: If samples effervesce upon addition of sodium bisulfate, than bisulfate cannot be used as a preservative. Another preservation option must be selected.
Note 4: If the purge temperature is >45ºC, bisulfate cannot be used as degradation of certain analytes (e.g. MTBE) may occur.
Note 5: 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.

Appendix A

Laboratory Requirements for Evaluation of

Tentatively Identified Compounds

Method 8260

A-1. Chromatographic Criteria

A-1.1 Initially include all of the non-target compounds that elute 30 seconds before the first target compound and 3 minutes after the elution of the last target compound. 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 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 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.

Appendix B

Low Detection 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 ground water samples collected from a GA area. This modification to the Connecticut DEP Recommended Protocol for Method 8260 may be used in place of Method 524.2 with the approval of the Connecticut DEP.

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 DEP Recommended Protocols for Method 8260. The upper linear range without dilution will vary with the laboratory, but is approximately 30 ug/L.

B-2 QC Requirements

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

B-2.2 The instrument must be calibrated at or below the reporting limit (RL) specified in Table 2B of this appendix.

B-2.3 A 25 mL purge volume must be used for all samples and standards.

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 tentatively identified compounds (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 1.2 of the DEP Recommended Protocols for Method 8260 and in Appendix A of this document.

Table 2B Reporting Limits for Method 8260 Low Detection Limit

Analyte	CAS Number	Aqueous RL, ug/L	Notes
Acetone	67641	5	
Acrylonitrile	107131	0.5	
Benzene	71432	0.5	
Bromobenzene	108861	0.5	
n-Butylbenzene	104518	0.5	
Sec-Butylbenzene	135988	0.5	
Tert-Butylbenzene	98066	0.5	
Bromodichloromethane	75274	0.5	
Bromoform	75252	0.5	
Bromomethane	74839	0.5	
2-Butanone (MEK)	78933	5	
Carbon Disulfide	75150	0.5	
Carbon Tetrachloride	56235	0.5	
Chlorobenzene	108907	0.5	
Chloroethane	75003	0.5	
Chloroform	67663	0.5	
Chloromethane	74873	0.5	
2-Chlorotoluene	95498	0.5	
4-Chlorotoluene	106434	0.5	
Dibromochloromethane	124481	0.5	
1,2-Dibromo-3-chloropropane (DBCP)	96128	Note 1	
1,2-Dibromoethane (EDB)	106934	Note 1	
Dibromomethane	74953	0.5	
1,2-Dichlorobenzene	95501	0.5	
1,3-Dichlorobenzene	541731	0.5	
1,4-Dichlorobenzene	106467	0.5	
trans-1,4-Dichloro-2-butene	110576	0.5	
Dichlorodifluoromethane	75718	0.5	
1,1-Dichloroethane	75343	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	

Table 2B Reporting Limits for Method 8260 Low Detection Limit (con't)

Analyte	CAS Number	Aqueous RL, ug/L	Notes
cis-1,3-Dichloropropene	10061015	0.5	
trans-1,3-Dichloropropene	10061026	0.5	
Ethylbenzene	100414	0.5	
Hexachlorobutadiene	87683	0.45	
2-Hexanone	591786	5	
Isopropylbenzene (Cumene)	98828	0.5	
4-Isopropyltoluene	99876	0.5	
Methylene Chloride	75092	0.5	
4-Methyl-2-pentanone (MIBK)	108101	5	
Methyl-tert-butylether (MTBE)	1634044	0.5	
Naphthalene	91203	0.5	
n-Propylbenzene	103651	0.5	
Styrene	100425	0.5	
1,1,1,2-Tetrachloroethane	630206	0.5	
1,1,2,2-Tetrachloroethane	79345	0.5	
Tetrachloroethene (Perc)	127184	0.5	
Tetrahydrofuran (THF)	109999	5	
Toluene	108883	0.5	
1,2,3-Trichlorobenzene	87616	0.5	
1,2,4-Trichlorobenzene	120821	0.5	
1,1,1-Trichloroethane	71556	0.5	
1,1,2-Trichloroethane	79005	0.5	
Trichloroethene (TCE)	79016	0.5	
Trichlorofluoromethane	75694	0.5	
1,2,3-Trichloropropane	96184	0.5	
Trichlorotrifluoroethane (Freon-113)	76131	0.5	
1,2,4-Trimethylbenzene	95636	0.5	
1,3,5-Trimethylbenzene	108678	0.5	
Vinyl Chloride	75014	0.5	
o-Xylene	95476	0.5	See 2
m-Xylene	108383	0.5	See 2
p-Xylene	106423	0.5	See 2

Notes:
1. These compounds require analysis by either Methods 504.1 or other method approved by the Commissioner to achieve the RSR limit in aqueous samples.
2. May be reported as total xylenes or any combination of the three isomers.