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LABORATORY SOP FOR EPA METHOD 8260B/624: VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE

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PURPOSE: This SOP is used to insure consistent operation of the GC/MS systems within the EPD GC/MS laboratory for EPA Method 8260B. This method and analysis are also used for 624 WPCP sample analysis. It is intended as a guide for the daily operation of these instruments as well as a reference for maintenance, troubleshooting, and quality control. Note: All footnote references are to Method 8260B sections, unless otherwise noted.

1. SCOPE AND APPLICATION

1.1 Method 8260B is used to determine the concentration of the volatile organic compounds in liquids, sediments, and a variety of multi-phase samples. The operating procedures described here are designed to meet or exceed the requirements for method 8260B. The following compounds are currently analyzed in the Georgia EPD laboratory:

Compound	CAS No.
Acetone	67-74-1
Acrylonitrile	107-13-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butyl benzene	104-51-8
sec-Butyl benzene	135-98-8
tert-Butyl benzene	98-06-6

Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Cyclohexane	00110-82-7
1,2-Dibromo-3-chloropropane	96-12-8
Dibromochloromethane	124-48-1
1,2-Dibromomethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
trans-1,4-Dichloro-2-butene	110-57-6
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichoropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone	591-78-6
Iodomethane	74-88-4
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Methyl acetate	0079-20-9
Methylene chloride	75-09-2
Methylcyclohexane	00108-87-2
4-Methyl-2-pentanone	108-10-1
Methyl tert-butyl ether (MTBE)	1634-04-4
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4

Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
1,1,2-Trichlorotrifluoroethane	00076-13-1
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl acetate	75-01-4
Vinyl chloride	75-01-4
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

1.2 The estimated quantitation limit for each compound is instrument dependent. Method 8260B is for use on a purge/trap with a gas chromatography/mass spectrometer.

1.3 Due to instrument configuration differences to achieve the best recovery for each analyte of interest, Method 8260B analytes are subdivided into the following categories:

1.3.1 8260B Water-Standard Method, Non-heated purge vessel

1.3.2 8260B Soil-Standard Method, Heated purge vessel

1.4 Samples are introduced into a gas chromatograph by a purge-and-trap method. Using an EST Centurion autosampler and EST Evolution purge-and-trap, sample components are purged with Helium and transferred to a sorbent trap. Upon completion of purging, the trap is heated and back flushed with helium to desorb the components onto the GC column. The GC column is temperature programmed to separate the components and introduce them to the Mass Spectrometer detector. Identification of target analytes is accomplished by mass spectral comparison with mass spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard to a seven point calibration curve.

2 Definitions

Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions. (See SOP reference 12.6)

3 Interferences

3.1 There is a potential for volatile organic compounds to be present on unclean glassware, syringes, purge-and-trap lines, and system Helium and liquid lines. Daily analysis of de-ionized, filtered water method blank is used to determine if there are any background interferences/contaminations in the instrumentation system before any samples are analyzed. If levels of contamination above the reporting limits are found, the problem must be corrected and another blank run to show that the system is free of contamination.

3.2 If interferences/contamination occurs before or during analysis, baking out the trap and GC oven may clean the system. Rinse blank should be run until the system baseline returns to

normal or target compounds are below reporting levels. The analytical column may need to be cut, the insert and septum changed. Gross contamination may require replacement parts, and possible complete dismantling of the system for a thorough cleaning of the autosampler, purge/trap, mass spec source, and sample transfer lines.

3.3 Interference of Methylene chloride may occur as it is the solvent used in the semi-volatiles laboratory and well as the extractions laboratory, and is an 8260B analyte. Glassware in the VOC room is not shared with other labs, and volumetric glassware used for standards are stored in a hot glassware oven until ready for use to keep them clean of possible VOC contamination.

3.4 Interference from Acetone or MEK in the Methanol used in the preparation of the waste sample for analysis.

3.5 To check for interferences for a waste method blank, or after a waste sample with high concentrations of compounds, a waste blank is prepared by adding 1 ml of purge and trap grade methanol into 50 ml of DI water.

3.6 To check for interferences for a water method blank or after a water sample with high concentrations of compounds, a DI water cleaning blank should be run after to prevent carryover into the next sample analysis.

3.7 To check for interferences for a soil method blank or after a soil sample with high concentrations of compounds, a soil cleaning blank should be run after to prevent carryover into the next sample analysis.

3.8 To verify any interferences during the 5035 soil sample preservation procedure, use two blank 40 ml VOC soil vials (5 ml of NaHSO4 solution with stir bar). Inside the hood, the first vial is never opened and label as "Closed blank." Also in the hood, the 2nd vial is labeled "Opened Blank" and is uncapped at the beginning of transferring soil samples from the EnCore device into the 40ml soil vial, then re-capped at the end. These two blank soil vials will help verify that no contamination occurred during soil sample preparation.

4 Safety

Refer to the Laboratory Safety, Chemical Hygiene and Fire Safety Plan, online revision, (SOP Reference 13.2).

5 Apparatus and Equipment

5.1 The GC/MS Lab uses 2 instrument systems for 8260 VOC analysis.

Table 5.1.1 GC/MS Laboratory Instrument Systems for 8260

System	Autosampler	Purge/Trap	Mass Spectrometer	Gas Chromatograph
GCMS13 VOC3	Centurion	EST Evolution	Agilent 5973	Agilent 6890
GCMS17 VOC4	Centurion	EST Evolution	Agilent 5975	Agilent 6890

5.2 A Centurion autosampler is used to automate the sample analysis procedure and can analyze up to 51 vials. The position, run sequence, and method editor for the Centurion are controlled with a built-in keypad. This is for instruments: GCMS14 VOC3 and GCMS15 VOC4.

5.3 The EST Evolution purge/trap controls the parameters for purge flow, trap, desorb, and baking. The method editor uses a display screen and mouse for inputting parameters.

5.4 The trap used for 8260 volatiles analysis is a Supelco Purge Trap K or equivalent for the EST Evolution. The trap **MUST** be preconditioned according to manufacturer's specifications when it is newly installed.

5.5 Purge/Trap parameters.

Table 5.5.1 Purge/Trap Parameters EST Evolution P/T

Parameter	**Value**
Line Temp, °C	130
Valve Temp, °C	130
MORT, °C	39
Purge Ready Temp, °C	32
Purge Temp, °C	36
Sample Heater, °C	On
Prepurge Time, Min.	0
Sample Prepurge Time, Min.	0
Sample Preheat Temp, °C	0
Sample Preheat Time, Min.	0
Purge Time, Min.	11
Drypurge Time, Min.	1.0
Purge Flow Rate, ml/min	40
GC Start Option	Desorb
GC Cycle Time, Min.	0
Cryo Focuser	Off
Desorb Preheat Temp, °C	245
Desorb Time, Min.	0.5
Desorb Temp, °C	250
Desorb Flow	Off
Desorb Preheat Delay, sec	12
Sample Drain	On
Bake Time, Min.	10
Trap Bake Temp, °C	250
MORT Bake Temp, °C	210
Bake Flow Rate ml/min	85
Sparger Bake Temp, °C	110
Centurion Line Temp, °C	110

5.6 Centurion Autosampler Parameters

5.6.1 Most parameters are valid for water and soil sample, however individual parameters for water or soil are listed separately in the table below as soil parameters are not valid for water samples.

Table 5.6.1.1 EST Centurion Autosampler	
Parameter	**Value**
Sample Loop Fill Time, sec	10
Loop Equilibrate Time, sec	5
Sample Transfer Time, sec	10
Need Rinse Time, sec	20
Need Sweep Time, sec	10
Sample Loop Rinse Time, sec	20
Sample Loop Sweep Time, sec	15
Sparger Rinse Cycles	On, 1
Sparger Rinse Transfer Time, sec	10
Sparger Rinse Drain Time, sec	220
Foam Rinse Cycles	3
Conc #1 Cycle Time	0
Water Heater Temp °C	85
Internal Standard Injection Size ul	5
Water Sample-Soil Water Addition	Off
Water Sample-Soil Preheat Time, min	0
Water Sample-Soil Preheat Temp, °C	0
Water Sample-Soil Purge Time, min	0
Water Sample-Soil Purge Temp °C	0
Water Sample-Soil Stirrer	Off
Water Sample-Soil Soil Valve Heater Temp °C	0
Water Sample-Soil Conc #1 Line Heater Temp °C	0
Water Sample-Soil Minimizer Bake Time	Off
Soil Sample-Needle Rinse Time	Off
Soil Sample-Sample Loop Rinse Time	Off
Soil Sample-Blank Water Addition	ON 1X
Soil Sample-Sample Preheat Time, min	0.5
Soil Sample-Sample Preheat Temp, °C	40
Soil Sample-Purge Time, min	11
Soil Sample-Purge Temp °C	40
Soil Sample-Stirrer	On, Medium
Soil Sample-Soil Valve Heater Temp °C	85
Soil Sample- Concentrator Line #1 Temp °C	140
Soil Sample-Minimizer Bake Time	On, 2min

5.7 GC Oven Temperature Parameters

Table 5.7.1 GC Oven Temperatures	
Parameter	GCMS14 VOC3 GCMS15 VOC4
Injection Source	Manual
Injection Location	Front
Mass Spec:	Enabled
Inlet B Temp, °C (Back)	Off
Detector B Temp,°C (or AUX)	200
Inlet A, °C (Front)	200
Oven Equip Time, Min.	0.5
Oven Max Temp, °C	260
Oven State:	On
Method BFB:	
Oven Initial Temp, °C	50
Oven Initial Time, Min.	0.5
Level 1 Rate, °C/Min.	50
Level 1 Final Temp, °C	200
Level 1 Final Time, Min.	2.0
Method 8260: Water & Soil	
Oven Initial Temp, °C	35
Oven Initial Time, Min.	4.0
Level 1 Rate, °C/Min.	8
Level 1 Final Temp, °C	120
Level 1 Final Time, Min.	0.0
Level 2 Rate, °C/Min.	20
Level 2 Final Temp, °C	200
Level 2 Final Time, Min.	2.0

5.7.1 Inlet and column parameters

Table 5.7.1.1 Inlet and Column Parameters	
Parameter	GCMS14 VOC3 GCMS15 VOC4
Constant Flow	1.0 ml/min
Column Type	DB624
Column Length, Meters	25.0
Column Diameter, Millimeters	0.2
Film Thickness, Micrometers	1.12
Gas:	Helium
Vacuum Compensation:	On

Split Flow: ml/Min, Split Ratio BFB: 8260:	 45.0 (50:1) 99.5 (100:1)
Inlet Purge B:	--
Inlet Purge A:	On
Splitless Injection:	No
Column:	DB624

5.8 Mass Spectrometer parameters

Table 5.8.1 Mass Spectrometer Parameters	
Parameter	GCMS14 VOC3 GCMS15 VOC4
Tune File Name:	bfb.u
Acquisition Mode:	Scan
Solvent Delay, Min.	3.5
EM Voltage:	Relative
EM Offset:	0
Scan Low Mass:	35
Scan High Mass:	550
Threshold:	150
Sampling No.	2

5.9 Preventive Maintenance is performed on each MS instrument every six months. Record services in the instrument maintenance logbook. Repairs must be recorded in the logbook. For any instrument problems preventing the analysis of samples, the instrument must be flagged with a "RED TAG" attached to the GC. See Section 8 of the Quality Assurance Plan for further instructions.

5.10 40ml clear open top septa vials

5.11 20ml clear open top septa vials

5.12 40ml clear open top septa vials with 0.5ml 1:1 HCl. ESS Item# 4050-0300-QC or equivalent.

5.13 40ml clear open top septa with 5ml of 20% Sodium Bisulfate solution and one 12mm X 4mm PTFE stir bar. ESS Item# 4038-5035-QC or equivalent.

5.14 Ottawa sand or equivalent for soil matix.

5.15 Purge and Trap grade Methanol.

5.16 Various sized gas tight syringes, from 1µL to 1,000µL.

5.17 Encore™ or equivalent soil samplers and T-handles.

5.18 High precision weighing analytical balance up to 4 decimal places, up to 100gm.

5.19 NIST standards purchased from certified sources, all COA filed in logbooks

5.20 Ultrapure filtered de-ionized water, with no detects above reporting levels.

6 Reagent and Standards

6.1 When certified standards are received, label the box and the certificate of analysis with date received and analyst initials. Place the box in the instructed storage (refrigerator, freezer, room temperature) and place the certificate of analysis in the 8260 standard certificates logbook under the appropriate section. If necessary, replace the old MSDS if a newer version is received in the MSDS logbook, under the 8260 section.

6.2 A GC/MS lab standards logbook is used to record all standards that are prepared for analysis for the GC/MS lab. In this logbook record the date, operators initials, manufacturer's name, lot number, expiration date, initial dilution, compounds in the standard and all calculations used to achieve final dilution.

6.3 A GC/MS lab reagent logbook is used to record all reagents used for standard analysis or for sample analysis. This can include Methanol, sand, etc.

6.4 All prepared working standard vials are labeled with the standard login number, standard name, analysis, concentration, and expiration date.

6.4.1 A direct transfer of standard is given an expiration date of 6 months from the date of transfer.

6.4.2 A standard which contains a mixture of standards prepared in the lab is given an expiration date of 6 months from the date of preparation.

6.4.3 All glassware used for standard preparation is stored in the glassware oven at 100ºC. (New glassware can be heated for one hour or more to ensure any residual volatiles are eliminated.)

6.4.4 All standards are prepared from NIST traceable certified stock solutions are diluted using certified purge-and-trap grade methanol in volumetric flasks.

6.4.5 After spiking and bringing to volume, the flask is inverted 3 times for mixing and immediately transferred to gas tight capped vials. Once capped the vial is further sealed with Teflon tape around the edge of the cap. The septum inside the gas tight valve must be changed when the cap is reused.

6.4.6 The Internal/Surrogate/Matrix Spike standards are loaded directly onto the Centurion's instrument standard vials.

6.4.7 All direct transfer and prepared mixed standards are stored in the standard refrigerator at <4ºC.

6.5 Organic free de-ionized filtered water is used to prepare the Method Blank, calibration curve standards, and LCS/LCSDup standards.

6.5.1 Organic residue grade methanol is used in general laboratory clean-up of glassware, autosampler valves, rinsing of instrument pathway after contamination problems.

6.5.2 Purge-and-trap grade methanol is used in all standards preparations and waste level sample preparations.

6.6 Internal Standard/Surrogate Spiking Solution

6.6.1 Internal standard/surrogate spiking solution is purchased at a concentration of 2,000µg/ml for each component in the mixture. The purchased standard is diluted as indicated below to produce a spiking solution for the Centurion autosampler. The Centurion autosampler auto-spikes a volume of 5µL into each 5ml sample when transferring the sample to the purge/trap instrument, the final concentration in the sample is 50µg/L.

Table 6.6.1.1 Centurion Internal Standard/Surrogate Solution			
Compound	Initial Concentration	Aliquot	Final Concentration
Chlorobenzene-d5(I.S.)	2000µg/ml	2,500µL	50µg/ml
1,4-Dichlorobenzene(I.S.)	2000µg/ml		50µg/ml
1,4-Difluorobenzene(I.S.)	2000µg/ml		50µg/ml
Pentafluorobenzene(I.S.)	2000µg/ml		50µg/ml
Bromofluorobenzene(S.S.)	2000µg/ml		50µg/ml
Dibromofluorobenzene(S.S.)	2000µg/ml		50µg/ml
Toluene-d8 (S.S.)	2000µg/ml		50µg/ml
1,2-Dichloroethane-d4(S.S.)	2000µg/ml		50µg/ml

Final volumetric of Int Std/Surg Spike solution in MEOH	10ml
Total Volume of Standard Aliquot	250µL
Total Volume of Purge/Trap grade MEOH added	9.750ml

6.7 A solution of Bromofluorobenzene (BFB) is prepared as a secondary dilution at a concentration of 25µg/ml. 2µL (50ng) is manually injected into the GC/MS system for the BFB tune method.

Table 6.7.1 BFB Standard Tune Solution			
Compound	Initial Concentration	Aliquot	Final Concentration
Bromofluorobenzene	2500µg/ml	50µL	25µg/ml

Final volumetric of BFB solution in MEOH	5ml
Total Volume of BFB Aliquot	50µL
Total Volume of Purge/Trap grade MEOH added	4.950ml

6.8 LCS/CCV/MDL/Calibration Curve 8260 Spiking Standards

6.8.1 Four different 8260 stock standard vials are prepared.

6.8.1 The standards are prepared in a volumetric flask then transferred to gas tight glass vials with septums and valves for storage.

6.8.1.1 The first standard is GAS, a vendor purchased 8260 gas compound vial is diluted to a high concentration stock standard.

6.8.1.2 The second standard is LIQUIDS, a vendor purchased 8260 liquid compound vial, plus a vendor purchased Acrolein/Acrylonitrile vial, plus a vendor purchased SOW compound vial are all diluted and combined to a high concentration stock standard. Acrolein is included in the manufacturer's compound formulation of the Acrolein/Acrylonitrile (A/A) standard, but Acrolein is not used for 8260.

6.8.1.3 The third standard is ADDITIONS, a vendor purchased 8260 Additions compound vial is diluted to a high concentration stock standard.

6.8.1.4 The fourth standard is LOW, it is an ultra low concentration standard prepared by diluting combining the GAS, LIQUID, and ADDITIONS stock standards into one vial.

6.8.2 8260 GAS Standard

Table 6.8.2.1 8260 Gas Standard			
Compound	**Initial Concentration**	**Aliquot**	**Final Concentration**
Dichlorodifluoromethane	2000µg/ml	625µL	250ug/ml
Chloromethane	2000µg/ml		250ug/ml
Vinyl chloride	2000µg/ml		250ug/ml
Bromomethane	2000µg/ml		250ug/ml
Chloroethane	2000µg/ml		250ug/ml

Final Volumetric used for 8260 Gas Std. in Methanol	5ml
Total Volume of Standard Aliquot	625µL
Total Volume of Purge/Trap grade Methanol added	4.375ml

6.8.3 8260 LIQUID Standard

Table 6.8.3.1 8260 Liquid Standard			
Compound	**Initial Concentration**	**Aliquot**	**Final Concentration**
Liquids purchased vial			
Trichlorofluoromethane	2000µg/ml	625µL	250µg/ml
1,1-Dichloroethene	2000µg/ml		250µg/ml
Iodomethane	2000µg/ml		250µg/ml
Carbon disulfide	2000µg/ml		250µg/ml
Methylene chloride	2000µg/ml		250µg/ml
trans-1,2-Dichloroethene	2000µg/ml		250µg/ml
Methyl tert-butyl ether	2000µg/ml		250µg/ml

1,1-Dichloroethane	2000µg/ml	250µg/ml
Vinyl acetate	2000µg/ml	250µg/ml
2,2-Dichloropropane	2000µg/ml	250µg/ml
cis-1,2-Dichloroethene	2000µg/ml	250µg/ml
Bromochloromethane	2000µg/ml	250µg/ml
Chloroform	2000µg/ml	250µg/ml
1,1,1-Trichloroethane	2000µg/ml	250µg/ml
Carbon tetrachloride	2000µg/ml	250µg/ml
1,1-Dichloropropene	2000µg/ml	250µg/ml
Benzene	2000µg/ml	250µg/ml
1,2-Dichloroethane	2000µg/ml	250µg/ml
Trichloroethene	2000µg/ml	250µg/ml
1,2-Dichloropropane	2000µg/ml	250µg/ml
Dibromomethane	2000µg/ml	250µg/ml
Bromodichloromethane	2000µg/ml	250µg/ml
cis-1,3-Dichloropropene	2000µg/ml	250µg/ml
Toluene	2000µg/ml	250µg/ml
trans-1,3-Dichloropropene	2000µg/ml	250µg/ml
1,1,2-Trichloroethane	2000µg/ml	250µg/ml
Tetrachloroethene	2000µg/ml	250µg/ml
1,3-Dichloropropane	2000µg/ml	250µg/ml
Dibromochloromethane	2000µg/ml	250µg/ml
1,2-Dibromoethane	2000µg/ml	250µg/ml
Chlorobenzene	2000µg/ml	250µg/ml
1,1,1,2-Tetrachloro-ethane	2000µg/ml	250µg/ml
Ethylbenzene	2000µg/ml	250µg/ml
p,m-Xylene	4000µg/ml	500µg/ml
o-Xylene	2000µg/ml	250µg/ml

Styrene	2000µg/ml		250µg/ml
Bromoform	2000µg/ml		250µg/ml
Isopropylbenzene	2000µg/ml		250µg/ml
Bromobenzene	2000µg/ml		250µg/ml
1,1,2,2-Tetrachloroethane	2000µg/ml		250µg/ml
1,2,3-Trichloropropane	2000µg/ml		250µg/ml
trans-1,4-Dichloro-2-butene	2000µg/ml		250µg/ml
N-Propylbenzene	2000µg/ml		250µg/ml
2-Chlorotoluene	2000µg/ml		250µg/ml
4-Chlorotoluene	2000µg/ml		250µg/ml
1,3,5-Trimethylbenzene	2000µg/ml		250µg/ml
tert-Butylbenzene	2000µg/ml		250µg/ml
1,2,4-Trimethylbenzene	2000µg/ml		250µg/ml
sec-Butylbenzene	2000µg/ml		250µg/ml
1,3-Dichlorobenzene	2000µg/ml		250µg/ml
p-Isopropyltoluene	2000µg/ml		250µg/ml
1,4-Dichlorobenzene	2000µg/ml		250µg/ml
1,2-Dichlorobenzene	2000µg/ml		250µg/ml
n-Butylbenzene	2000µg/ml		250µg/ml
1,2-Dibromo-3-chloropropane	2000µg/ml		250µg/ml
1,2,4-Trichlorobenzene	2000µg/ml		250µg/ml
Hexachlorobutadiene	2000µg/ml		250µg/ml
Naphthalene	2000µg/ml		250µg/ml
1,2,3-Trichlorobenzene	2000µg/ml		250µg/ml
Acrolein/Acrylonitrile purchased vial			
Acrylonitrile	10000µg/ml	1000µL	2000µg/ml
Acrolein	10000µg/ml		2000µg/ml
SOW purchased vial			

Cyclohexane	2000µg/ml	625µL	250µg/ml
Methylcyclohexane	2000µg/ml		250µg/ml
Methyl acetate	2000µg/ml		250µg/ml
1,1,2-Tetrachloro-1,2,2-trifluoroethane	2000µg/ml		250µg/ml

Final Volumetric used for 8260 Liquid Std. in Methanol	5ml
Total Volume of Standard Aliquot	2.250mL
Total Volume of Purge/Trap grade Methanol added	2.750ml

6.8.4 8260 ADDITIONS Standard

Table 6.8.4.1 8260 Additions Standard

Compound	Initial Concentration	Aliquot	Final Concentration
Vinyl acetate	2000µg/ml	625µL	250µg/ml
Acetone	2000µg/ml		250µg/ml
2-Butanone	2000µg/ml		250µg/ml
2-Hexanone	2000µg/ml		250µg/ml
4-Methyl-2-pentanone	2000µg/ml		250µg/ml
Carbon disulfide	2000µg/ml		250µg/ml
Iodomethane	2000µg/ml		250µg/ml
2-Chloroethyl-vinylether	2000µg/ml		250µg/ml

Final Volumetric used for 8260 Add. Std. in Methanol	5ml
Total Volume of Standard Aliquot	0.625mL
Total Volume of Purge/Trap grade Methanol added	4.375ml

6.8.5 8260 Low Level Soil Standard Vial used for Soil Calibration Curves

Table 6.8.5.1 8260 Low Level Standard

Prepared Stock Standard	Concentration	Aliquot	Final Concentration
Gases	250µg/ml	100µL	5µg/ml
Liquids	250µg/ml (A/A@ 2000µg/ml)	100µL	5µg/ml (A/A@40µg/ml)
Additions	250µg/ml	100µL	5µg/ml

Final Volumetric used for 8260 low level soil std. in Methanol	5ml
Total Volume of Standard Aliquots	0.3mL
Total Volume of Purge/Trap grade Methanol added	4.7ml

6.9 ICV/Second Source standards

6.9.1 A second set of standards must be purchased from a different manufacturer or from a different lot number if the same manufacturer. These standards are prepared in the same fashion and concentrations as the 8260 primary standards listed above. The ICV is analyzed after each calibration curve to verify the concentrations of the performance analytes (CCCs and SPCCs) meet method 8260 QC objectives.

7 Sample Collection

Refer to Chapter 5 of the Georgia EPD Laboratory Quality Assurance Manual for Sample Container, Sample Preservation, and Sample Holding Time for Water Samples. Requirements for Soil Samples are located in the Georgia EPD Laboratory SOP for EPA Method 5035.

7.1 Aqueous Sample Collection

The EPD Laboratory provides pre-preserved (1:1 HCl) 40 ml vials for the collection of aqueous samples. 4 vials are required for each sample submitted for analysis. Collectors should fill the vial to the top minimizing the amount of air bubble in the vial after the top is placed on the vial. Care should also be taken to avoid washing the HCl preservative out of the vial. Samples should be placed on ice as soon as possible after collection and cooled to ≤ 6°C (but not frozen). Holding time for properly preserved (pH<2) samples is 14 days.

7.2 Soil Sample Collection

The EPD Laboratory provides Encore™ samplers for the collection of soil samples for volatile organic analysis. Four Encore™ samplers and one 4oz. glass bottle are required for each sample. Samples should be placed on ice as soon as possible after collection and cooled to ≤ 6°C (but not frozen). Samples must be preserved in 5ml NaHSO4 solution vials within 48 hours of collection time. The Laboratory must be notified in advance of sample delivery. Detailed sample collection instructions and tools are provided with each soil sampling kit.

8 Calibration

8.1 Remove the prepared Gas, Liquid, and Additions (and low level standard if needed) standard vials from the refrigerator and allow them to equilibrate to room temperature.

8.1.1 *BFB Mass Spectrometer Tune*

The mass spectrometer must be initially hardware tuned to be able to meet the BFB M/Z abundance criteria in method 8260B. The ChemStation software has an automatic BFB tune program for the mass spectrometer, after running the program the tune parameters are saved to BFB.U.

8.1.2 In ChemStation run the BFB data method and direct inject 2 µl of the BFB standard into the GC and press the "Start" button on the GC. A passing tune is valid for 12 hrs.

8.1.3 The resulting BFB peak must meet the abundances in table 8.1.3.1. Only the 3 scans at the apex may be used, background subtraction is allowed. The Chemstation software will automatically generate a BFB tune evaluation if the "Evaluate BFB" button is selected.

Table 8.1.3.1 – Mass Spectrometer Tune Criteria

Mass (M/Z)	Relative Abundance Criteria
50	15.0 to 40.0 percent of m/e 95
75	30.0 to 80.0 percent of m/e 95
95	base peak, 100 percent relative abundance
96	5.0 to 9.0 percent of m/e 95
173	less than 2.0 percent of m/e 174
174	>50.0 percent of m/e 95
175	5.0 to 9.0 percent of m/e 174
176	>95.0 but < 101.0 percent of m/e 174
177	5.0 to 9.0 percent of m/e 176

8.2 *Calibration Standards*

8.2.1 *Soil Curve* calibration levels are prepared based on a 5.0 g sample (0.005 kg), the curve standards are added directly to the purchased pre-preserved NaHSO4 soil vials.

8.2.2 *Water Curve* calibration levels are based on a 5.0 ml sample and are prepared using a 50.0 ml volumetric flask with DI water.

8.2.3 *Waste Curve* calibration levels are based on a 5.0 ml purged subsample of the waste which will contain 0.1 g of the waste in the sparging tube.

8.2.3.1 The waste subsample is prepared by diluting 5.0 g of the waste with 5.0 ml MeOH in a 20ml vial. 1.0 ml of this dilution is pipetted into a 50.0 ml volumetric flask, then filled to level with DI water.

8.2.3.2 The calculations for the waste subsample are:
5.0g (waste) /5.0ml (Methanol) = 1.0g/ml (waste concentration in dilution vial)
1.0g/ml (dilution vial) * 1.0ml (pipetted) = 1g (waste weight in pipette)
1g (waste weight)/50.0ml (volumetric flask) = 0.02g/ml (waste concentration in flask)
0.02 g/ml (waste conc.) * 5.0ml (purged sample size in sparger) = 0.1g (waste weight purged)

8.2.3.3 For the waste curve concentration levels, the amount of standard in the sparger is divided by the amount of waste in the sparger.

8.2.3.4 Waste calibration curve calculation example:
Curve point standard prepared at a concentration of 5ug/L.

5ml of curve point is transferred to the sparge tube = 0.025ug of standard
Curve based on 0.1gm of waste in sparge tube (after dilutions as discussed above)
Curve point is:
0.025ug standard/ 0.1gm of waste = 0.000025mg/0.0001Kg = 0.25mg/Kg

8.2.4 The water calibration curve consists of the calibration standards at the following concentrations, Acylonitrile concentrations are also listed due to their higher concentration in the Liquid stock standard, *Acrolein* is not used in the 8260 water curve.

Table 8.2.4.1 8260 Water Calibration Curve Vials

Gas Stock 250µg/ml Aliquot	Liquid Stock 250µg/ml Aliquot	Additions Stock 250µg/ml Aliquot	Volumetric Flask Volume	Final Concentration
0.4 µL	0.4 µL	0.4 µL	50 ml	2µg/L (Acrylonitrile@16µg/L)
1.0 µL	1.0 µL	1.0 µL	50 ml	5µg/L (Acrylonitrile @40µg/L)
10.0 µL	10.0 µL	10.0 µL	50 ml	50µg/L (Acrylonitrile @400µg/L)
20.0 µL	20.0 µL	20.0 µL	50 ml	100µg/L (Acrylonitrile @800µg/L)
30.0 µL	30.0 µL	30.0 µL	50 ml	150µg/L (Acrylonitrile @1200µg/L)
40.0 µL	40.0 µL	40.0 µL	50 ml	200µg/L (Acrylonitrile @1600µg/L)
80.0 µL	80.0 µL	80.0 µL	50 ml	400µg/L (Acrylonitrile @3200µg/L)

8.2.4.1 The Chemstation software 8260 water calibration curve will begin all 8260 compounds at 5µg/L **except** for the following compounds listed in table 8.2.4.1.1, the list has the individual calibration curve starting points for these compounds. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

Table 8.2.4.1.1 Water Calibration Curve starting point exceptions

Compound	Concentration
Vinyl Chloride	2ug/L
Acrylonitrile	40ug/L
Acetone	50ug/L
Vinyl Acetate	50ug/L
2-Butanone	50ug/L
4-Methyl-2-Pentanone	50ug/L
2-Hexanone	50ug/L

8.2.5 The soil calibration curve consists of the calibration standards at the following concentrations

Table 8.2.5.1 8260 Soil Calibration Curve Vials					
Low Level 5µg/ml Aliquot	**Gas Stock 250µg/ml Aliquot**	**Liquid Stock 250µg/ml Aliquot**	**Additions Stock 250µg/ml Aliquot**	**Soil Weight**	**Final Concentration**
2.0 µL	0	0	0	5.0 gm	2µg/Kg
5.0 µL	0	0	0	5.0 gm	5µg/Kg
0	1.0 µL	1.0 µL	1.0 µL	5.0 gm	50µg/Kg
0	2.0 µL	2.0 µL	2.0 µL	5.0 gm	100µg/Kg
0	3.0 µL	3.0 µL	3.0 µL	5.0 gm	150µg/Kg
0	4.0 µL	4.0 µL	4.0 µL	5.0 gm	200µg/Kg
0	8.0 µL	8.0 µL	8.0 µL	5.0 gm	400µg/Kg

8.2.5.1 Compounds *Trans-1,4-Dichloro-2-butene* and *Acrolein* and *Acrylonitrile* in the standard mixes listed in Table 6.8.3.1 but are not used for the soil calibration curve.

8.2.5.2 The soil calibration curve will begin all 8260 compounds at 5µg/L except for the compounds listed in table 8.2.5.2.1 which begin the lowest curve point at a different concentration. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

Table 8.2.5.4.1 Soil Calibration Curve starting point exceptions	
Compound	**Concentration**
Acetone	50µg/Kg
2-Butanone	50µg/Kg
4-Methyl-2-Pentanone	50µg/Kg
2-Hexanone	50µg/Kg
Vinyl Acetate	50µg/Kg

8.2.6 The waste calibration curve consists of the calibration standards at the following concentrations in table 8.2.6.2.1. Methanol is added to each curve level to match the amount of Methanol in the prepared diluted waste sample that is purged.

8.2.6.1 Compounds *Trans-1,4-Dichloro-2-butene* and *Acrolein* and *Acrylonitrile* are not used for the Waste Calibration Curve

8.2.6.2 The MDL vial is prepared at 0.25mg/Kg except for the compounds listed in Table 8.2.6.3.1, which will at the lowest calibration point. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

Table 8.2.6.2.1 8260 Waste Calibration Curve Vials					
Gas Stock 250µg/ml Aliquot	**Liquid Stock 250µg/ml Aliquot**	**Additions Stock 250µg/ml Aliquot**	**Methanol Added**	**Volumetric Flask Volume**	**Final Concentration**
1.0 µL	1.0 µL	1.0 µL	997µL	50 ml	0.25mg/kg
2.0 µL	2.0 µL	2.0 µL	994µL	50 ml	0.5mg/kg
10.0 µL	10.0 µL	10.0 µL	970 µL	50ml	2.5mg/kg
20.0 µL	20.0 µL	20.0 µL	940µL	50 ml	5.0mg/kg
30.0 µL	30.0 µL	30.0 µL	910µL	50 ml	7.5mg/kg
40.0 µL	40.0 µL	40.0 µL	880µL	50 ml	10.0mg/kg
80.0 µL	80.0 µL	80.0 µL	760µL	50 ml	20.0mg/kg

8.2.6.3 The lowest point of the curve begins at 0.25mg/Kg except for the compounds listed in table 8.2.5.4.1. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

Table 8.2.6.3.1 Waste Curve Calibration Curve starting point exceptions	
Compound	Concentration
Acetone	2.5mg/Kg
2-Butanone	2.5mg/Kg
4-Methyl-2-Pentanone	2.5mg/Kg
2-Hexanone	2.5mg/Kg
Vinyl Acetate	2.5mg/Kg

8.3 *Building the Calibration Curve using the Chemstation software*

8.3.1 After passing the BFB Tune, the calibration curve level standard vials are analyzed on the instrument using Chemstation data acquisition software.

8.3.2 Each matrix, water, soil, and waste curve is uses its own separate matrix data acquisition method and data analysis method.

8.3.2 After the curve vials are analyzed and edited and reviewed for peak shape and retention time, the results for each compound are loaded into the calibration curve. The curve method name is saved as the date the curve is prepared along with the letters of the matrix used.

8.3.2.1 NOTE: Due to the EPA MDL update rule, in Chemstation not all compounds will have a value at the lowest concentration level of the curve. The compounds that have exceptions to their curve starting point will have an empty box at the lowest concentration point of the curve.

8.3.2.1 The compounds that have exceptions for their curve starting point will have their lowest curve concentration value be at a higher concentration value than the majority of compounds curve starting point.

8.3.3 The ChemStation data analysis software will calculate a Response Factor for each compound for each calibration level and an average for all levels that can be printed out.

8.3.4 The software will also calculate a percent Relative Standard Deviation for each compound. This percent Relative Standard Deviation (%RSD) should be less than 15% for each compound.

8.3.5 For any target compound (excluding CCC compounds) a %RSD greater than 15% then a non-linear or linear regression can be used.

8.3.5.1 If a Quadratic or Linear fit is used, must be $r \geq 0.990$.

8.3.6 System Performance Check Compounds(SPCCs) Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane and Chlorobenzene must have a minimum average response factor for the initial curve of 0.10.

8.3.7 System Performance Check Compounds(SPCCs) Chlorobenzene, and 1,1,2,2-Tetrachloroethane, must have a minimum average response factor for the initial curve of 0.30.

8.3.8 For each Calibration Check Compound(CCCs), Vinyl chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene and Ethylbenzene. The RSD must be <30% RSD.

8.3.9 ICV, the Second Source standard, must be run after each calibration curve for accuracy verification. This standard should be run at the same level as the LCS. The percent deviation should be ± 30% for all performance compounds (CCC and SPCC).

8.4 *Calibration Curve Verification (CCV)*

8.4.1 The mass spec tune must be verified by injecting 50 ng of BFB tune and checking ion abundances against the tune acceptance criteria. BFB must be run first in any sequence and is valid for 12 hours.

8.4.1.2 If sample analysis will run past 12 hours, the Tune must be verified again for each additional 12-hour period.

8.4.2 After BFB, the CCV is run by analyzing a full 8260 standard at the midpoint of the curve, a concentration of 50 µg is used for water, soil, and waste. It is valid for 20 field samples in a batch/shift. If more than 20 are analyzed, a new CCV and batch must be created.

8.4.3 The System Performance Check Compounds (SPCCs) must meet the same criteria for a minimum relative response factor as described above for the calibration curve.

8.4.4 CCC compounds in the CCV sample must have a percent deviation of <20%.

8.4.4.1 Compounds other than CCC should have a deviation of <20%. However, it is permissible to have 5% of the compounds fail high (>20% deviation) if there are no detects above the MRL for the compound in any samples of the batch.

8.4.5 All compounds (other than CCC listed above) must have a percent deviation of <20%.

8.4.5.1 If any compound fails low (<20% deviation), a new CCV can be prepared and analyzed.

8.4.5.2 If the source of the CCV failure cannot be identified and the CCV deviation still fails low (<20% deviation), a new eight-point calibration curve must be generated.

8.4.5.3 If a CCV compound fails low (<20% deviation) sample analysis cannot continue without re-running the CCV, building a new calibration curve, or inspecting the instruments for possible corrective action or repair.

8.4.5.1 Any linear fit or quadratic fit compounds must have a deviation of <20% from the nominal value of the CCV for that compound.

8.4.6 CCV evaluation using ChemStation software, make sure to use the same method as the calibration curve for CCV evaluation. Go to Contcal and then Evaluate Datafile.

8.4.6.1 Display or print the evaluation, the acceptance criteria for the SPCCs and the CCCs are already entered into the software on page 2 for these compounds. **C** or **P** to the left of a compound indicates CCC or SPCC respectively. The bottom of the report will display how many SPCCs and CCCs fail QC criteria. This should state "0" for both.

8.4.7 Internal standard retention times cannot drift by more than 30 seconds from one Calibration Check to the next (every 12 hours). The EICP area for internal standards should be between 50 to 200% from the mid-point standard level of the most recent initial calibration sequence. If either of these criteria is not met, a problem with GC/MS is indicated and the problem must be corrected and verified by another standard analysis before any sample analysis.

9 Quality Control

9.1 *BFB,* the GC/MS system must meet BFB criteria every 12 hour shift.

9.1.1 20 field samples max per QC batch, does not include QC samples.

9.1.2 If 20 sample analysis batch runs over 12 hrs, new BFB must be analyzed to extend 12 hrs.

9.2 *Initial Calibration Curve* must meet QC criteria

9.2.1 The software will calculate a percent Relative Standard Deviation for each compound. This percent Relative Standard Deviation (%RSD) should be less than 15% for each compound.

9.2.2 For any target compound (excluding CCC compounds) if %RSD greater than 15% then a non-linear or linear regression may be used.

9.2.3 If a Quadratic or Linear fit is used, must be $r \geq 0.990$.

9.2.4 System Performance Check Compounds(SPCCs) Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane and Chlorobenzene minimum average response factors for the initial curve of 0.10.

9.2.5 System Performance Check Compounds(SPCCs) Chlorobenzene, and 1,1,2,2-Tetrachloroethane, minimum average response factors for the initial curve of 0.30.

9.2.6 ICV, second source verification of the instrument with each new curve built. The percent deviation should be ± 30% for all performance compounds (CCC and SPCC compounds must be ± 30%).

9.3 *CCV*, daily/20 samples max must meet the SPCC and CCC criteria for each 12 hour shift (run after BFB).

9.3.1 The System Performance Check Compounds (SPCCs) must meet the same criteria for a minimum relative response factor as described above for the calibration curve.

9.3.2 CCC compounds must have a percent deviation of ±20%.

9.3.3 All compounds (other than CCC) must have a percent deviation of ±20%, if any compound fails low (<20% deviation), a new CCV can be prepared and analyzed. However if the source of the failure cannot be identified and the CCV deviation still fails low (<20% deviation), a new seven point calibration curve must be generated. If any CCV compound fails low (<20% deviation) sample analysis cannot continue.

9.3.4 CCV (other than CCC) compounds should have a deviation of <20%. It is permissible to have 5% of the compounds fail high (>20% deviation) and if there are no hit for these compounds in any samples of the batch.

9.3.5 Any linear fit or quadratic fit compounds must have a deviation of <20% from the nominal value of the CCV for that compound.

9.4 *Internal standards* are Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d5 and 1,4-Dichlorobenzene-d4. Area counts of the internal standard peaks should be between 50 - 200% of the areas of the internal standards in the mid-point standard of the most recent initial calibration analysis.

9.5 *Surrogates* are Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8 and Bromofluorobenzene. Limits are set through annual charting.

9.6 *Method Blank* is required to run on the instrument system for each matrix for each batch. The

purpose of a blank is to verify that the instrument and all reagents associated with an analysis are free from contamination (no compounds >reporting limits).

9.6.1 A water Method Blank is a 40 ml sample vial is filled with deionized water.

9.6.2 A soil Method Blank is a purchased 40 ml VOC vial pre-preserved with 5 ml of NaHSO4 solution.

9.6.3 A waste Method Blank is 1 ml of purge and trap grade methanol added to a 50 ml volumetric flask and deionized water added to level, then transferred to a clean 40 ml sample vial.

9.7 *Laboratory Control Spike,* LCS, (Laboratory Fortified Blank) and Laboratory Control Spike Duplicates (LCSD), consists of 50 µg standards each, that are analyzed sequentially at the beginning of each sample batch (after the CCV). The LCS/LCSD spike compounds are 1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene. The recoveries and precisions of these compounds must meet quality control limits.

9.7.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are utilized to determine quality control limits that are affected by sample matrix interference. The spike compounds are 1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene with a concentration of 50 µg. The recovery and precision of these compounds should meet quality control limits.

9.8 *Charting* for water, soil, waste samples for the LCS/LCSD, Surrogates, and MS/MSD for upper and lower recovery control limits are performed twice per year. However the Appendix A control limits are updated only annually. These upper and lower control limits are based on 500 data points or up to 48 months of data may be used if less than 20 points are available.

9.8.1 If the lower recovery control limit for any compound is less than 10%, then the lower control limit will be set at 10%.

9.8.2 If the calculated upper recovery control limit for any compound is less than 100%, then the upper control limit will be set at 130%.

9.8.3 If the upper recovery control limit is >200%, the upper limit will be set at 200%.

9.8.4 If the lower recovery control limit is greater than 70%, i.e 75%, the limit will be set to 70%.

9.8.5 If the upper recovery control limit is less than 130%, i.e 125%, the limit will be set to 130%.

9.8.6 The precision for LCS/LCSD and MS/MSD are static at: Water ≤30%RPD, Soil ≤ 40%RPD, and Waste ≤50%RPD.

9.9 *MDL* (method detection limit) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The actual MDL varies depending on instrument and matrix.

9.9.1 The MDL must be determined for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.9.2 Method Detection Limit Study for all analytes must be performed initially and then minimally once per year or after major instrument repairs or changes to procedures. There are 2 ways to perform the MDL, the first is with 7 samples and 7 blanks over 3 separate non-consecutive days, and the second is the preferred method with the MDL run as a continuous format.

9.9.3 The 7 MDL samples study is performed by preparing 7 spiked vials, MDL_{Spike} , spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDL_{Blank}. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there is a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blank. MDL spikes and blanks must contain any preservative used in the analysis of samples such as sodium bisulfate used for soil samples.

9.9.4 Under the EPA MDL update rule, the preferred MDL study method is a continuous format

MDL study and is performed where one vial is spiked as an MDL_{Spike} , at the lowest point of the calibration curve for each compound and analyzed with every batch of samples along with the method blank vial as an MDL_{Blank}.

9.9.5 The results of the MDL_{Spike} and MDL_{Blank} will be entered into Labworks using the Method Blank test code for the matrix (water, soil, or waste). The MDL result will be entered using the $ML826([W]water, [S]soil, or [T]waste). The MDL Spiked Amount will be entered into $MA826(water, soil, or waste). The instrument used for the MDL and Blank analysis will selected using INSTR-826(instrument).

9.9.6 MDL study must be performed on a yearly basis, before the MDL for the instrument expires. There is an MDL SOP that must be followed.

9.10 Initial capability demonstration of the analyst's ability to produce acceptable recovery, accuracy, and precision the following operation must be performed for each matrix before an analyst can sample run analysis. Results must meet reporting limits listed in Appendix A, Table A.1.

9.10.1 Analysts must also perform a Continuing Demonstration (CDF) every six months to prove the analyst's ability to produce acceptable recovery, accuracy, and precision for each matrix. Results must meet limits listed in Appendix A, Table A.1.

9.10.2 For the 4 CDF replicates, the midpoint of the curve (50 µg) for each matrix must be analyzed using the appropriate data analysis method.

9.11 All printed paperwork is saved for 10 years

9.11.1 All sequences are printed brief and detailed, the brief format is saved in a sequence logbook.

9.11.2 All calibration curve related paperwork is printed and saved in a file folder and stored.

9.11.3 All QC attached to a batch is stored in the batch file folder (BFB tune, CCV, Method Blank, MDL analysis, sequence pages, Int Std Area reports, sample reports, dilution pages, extraction sheets, sample preparation sheets, etc.)

10 Procedure

10.1 When a sample is received in the laboratory, it is checked against the chain of custody paperwork to ensure that a 8260 test is ordered for the sample.

10.2 Water samples are stored in the sample refrigerator (<4ºC but not frozen) immediately upon receipt.

10.2 Place waste samples in individual Ziploc sample bags to prevent sample fumes from migrating into the refrigerator and possibly contaminating other samples.

10.3 Prepare soil samples according to SOP for method 5035. Low level samples are preserved in 40 ml vials containing 5 ml of sodium bisulfate solution with a magnetic stir bar and high level samples are preserved in 20 ml vials containing 5 ml of methanol. Samples should then be placed in the sample refrigerator (<4ºC but not frozen) immediately until it is ready to be analyzed to minimize loss of volatile compounds.

10.3.1 Preserved low level soil vials and high level Methanol extraction vials have a holding time of 14 days from the preservation date.

10.4 Always wear appropriate protective wear such as labcoat, gloves, eye protection, and prepare all samples under the hood.

10.5 *GCMS Instrument Sample Analysis*

10.5.1 All samples and standard solutions must be at ambient temperature before analyzing.

10.5.2 BFB and CCV must meet QC prior to sample analysis.

10.5.2.1 If a valid, new calibration curve is built, no CCV is needed for that 12hr shift.

10.5.3 Take only one 5ml aliquot per vial, if a reanalysis is needed to confirm an unusual detected compound, use the second vial. The second sample vial can also be used for a dilution in cases of detects over the calibration curve upper limits or high matrix interferences.

10.5.4 The Centurion will automatically add 5 µl internal and surrogate standard.

10.5.5 For matrix spike sample, the Centurion automatically adds 5 µl of matrix spike mixture.

10.5.6 If a dilution is performed on a sample, the final dilution results need to be multiplied by the dilution factor to calculate the final concentration of the target compound.

10.6 *Water Sample*

10.6.1 Allow water samples to come to ambient temperature before analysis.

10.6.2 Set the 40ml sample vial in the Centurion, a 5 ml aliquot of water sample is withdrawn from sample vial through the syringe of Centurion autosampler. Use the water method for the Centurion.

10.6.3 The Centurion uses a 5 µl injection loop to add the internal and surrogate standard mixture automatically to the 5 ml sample and yields a final concentration of 50 µg/L.

10.6.3.1 For Matrix Spike/Matrix Spike Duplicate samples the Centurion also adds 5 µl of matrix spike standard mixture to a 5 ml sample and yields a final concentration of 50 µg/L.

10.6.4 The Centurion will transfer the sample to the purge vessel on the front of the purge/trap concentrator. The purging will run for 11 minutes. The GC will signal ready to the purge/trap to begin to desorb for 4 minutes. After desorbing, the purge/trap will go to the bake cycle. After baking, the purge/trap will cool off the trap and the cycle will begin again for the next sample.

10.6.5 After the samples are finished on the sequence the MDL vials will be run, for 8260 water MDL vials will be made using the following guidelines.

10.6.5.1 The water MDL vials are prepared at the concentration listed in table 10.6.5.2.1. This follows the EPA MDL update rule where the MDL mixture is prepared at the lowest calibration curve starting point of each compound.

10.6.5.2 For the 8260 Water MDL, two vials are made (Vial 1 & 2) using Table 10.6.5.2.1, making the MDL vials using the table allows for all the compounds to start at their respective lowest calibration point.

Table 10.6.5.2.1 8260 Water MDL Vials

MDL Vial	Gas Vial 250ug/L aliquot	Liquid Vial 250ug/L aliquot	Addition Vial 250ug/L aliquot	Volumetric Flask Volume	Final Concentration
Vial #1	Add 1.0uL	Add 1.0uL	Add 1.0uL	50ml	5ug/L
Vial #2	Add 0.4uL		Add 10.0uL	50ml	2ug/L (for Vinyl Chloride) 50ug/L (for Exceptions)

10.6.5.3 Using the data obtained, in labworks upload the 5ug/L MDL and manually replace the results for the exceptions, which can be viewed at Table 8.2.4.1.1, from the other (Vial 2) MDL vial's data.

10.6.5.4 Uploading the MDL data this way will allow for the correct starting calibration point for each compound to be shown.

10.7 *Soil Sample*

10.7.1 Allow soil samples to come to ambient temperature before analysis.

10.7.2 Shake the low level soil sample vials to eliminate precipitation and settlement of soil to the

bottom of vial and load into the Centurion tray. Use the soil method for the Centurion.

10.7.3 The Centurion will auto-inject a 10 ml aliquot of DI organic free water from the Centurion water reservoir bottle into the soil vial.

10.7.4 The Centurion will add 5µl of internal and surrogate standard mixture through the 5 µl injection loop with the 10 ml organic free water.

10.7.5 If the matrix spike or matrix spike duplicate is used, the Centurion will also add 5 µl of matrix spike standard to the 10 ml organic free water.

10.7.6 The Centurion will preheat the soil vial at 40º C for 1 minute and then purge the vial itself for 11 minutes at 40º C with the stirring bar motor tuned on**.**

10.7.7 After purging for 11min, the purge/trap will desorb and bake as described earlier.

10.7.8 For high level soil samples the Encore soil sample weighed out into 5 ml with Methanol is used.

10.7.8.1 After the samples are run the MDL vials will be run after, for 8260 Soil MDL vials will be made using the following guidelines.

10.7.8.2 The MDL vial is prepared at 5.0µg/Kg except for the compounds listed in Table 8.2.4.1.1, which will at the lowest calibration point. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

10.7.8.3 The 8260 Soil uses three MDL vials, MDL vials are made using Table 10.7.8.3.1, making the MDL vials using the table allows for all the compounds to start at their respective lowest calibration point.

Table 10.7.8.3.1 8260 Soil MDL Vials

MDL Vial	Low Vial Std 5ug/mL aliquot	Addition Vial 250ug/L aliquot	Final Concentration
Vial #1	2.0uL		2.0ug/Kg (Vinyl Chloride)
Vial #2	5.0uL		5.0ug/Kg
Vial #3		1.0uL	50.0ug/Kg (Exceptions)

10.7.8.4 Using the data obtained, in labworks upload the 5.0ug/kg MDL and manually replace the results for the exception compounds, which can be viewed at Table 8.2.4.1.1, from the other (Vial 2) MDL vials' data.

10.7.8.5 Uploading the MDL data this way will allow for the correct starting calibration point for each compound to be shown.

10.7.8.6 For high level analysis, withdraw 100 µl and spike into a prepared soil NaHSO4 solution vial.

10.7.8.7 Use the steps as described above for a normal soil sample.

10.7.8.8 Do not add more than 100 µl of the high level extracted/methanol solution to the NaHSO4 vial as this is too high a level of methanol for the instrument to handle.

10.7.8.9 The results will be at a 50x dilution.

10.7.9 Soil samples are reported as % Dry Solids, a correction factor must be determined since field samples are not exactly 100% dry solids and exactly at 5gm of weight.

10.7.9.1 For all field samples the reporting limits and detected compounds must be multiplied by the correction factor. Results and reporting limits for soils should be multiplied by the dilution factor before reporting.

$$\frac{5\text{ g}}{\text{Weight of Sample} \times \%\text{ Dry Solids}} = \%\text{ Dry Solids Correction Factor}$$

10.8 *Waste Sample*

10.8.1 Allow waste sample to come to ambient temperature before analysis.

10.8.2 Weigh out 5 g of sample into a 20ml clean dry glass vial.

10.8.3 Add 5 ml of purge-and-trap grade Methanol, close the vial, and shake for 2 minutes.

10.8.4 To prepare for analysis, withdraw 1 ml of this Methanol extract from the vial and transfer it to a 50 ml volumetric flask, bring up to 50ml with DI organic free water.

10.8.5 Invert the flask 3 times, then fill a 40ml vial with the sample.

10.8.6 Prepare a 40ml vial Methanol blank (1ml Methanol in volumetric 50ml using DI water) to check for contamination or interference from the purge/trap Methanol.

10.8.7 The sample will follow the steps described above for a normal water sample analysis.

10.8.8 After the samples are run the MDL vials will be run after, for 8260 Soil MDL vials will be made using the following guidelines.

10.8.8.1 The MDL vial is prepared at 0.25mg/Kg except for the compounds listed in Table 8.2.6.3.1, which will at the lowest calibration point. Per EPA MDL update rule, the MDL mixture is always prepared at the lowest calibration curve point of each compound.

10.8.8.2 The 8260 Waste uses three MDL vials, MDL vials are made using Table 10.8.8.2.1, making the MDL vials using the table allows for all the compounds to start at their respective lowest calibration point.

Table 10.7.8.3.1 8260 Waste MDL Vials

MDL Vial	Gas Vial 250ug/L aliquot	Liquid Vial 250ug/L aliquot	Addition Vial 250ug/L aliquot	Methanol Added	Volumetric Flask Volume	Final Concentration
Vial 1	1.0uL	1.0uL	1.0uL	997uL	50ml	0.5mg/Kg
Vial 2	0.4uL		10.0uL	989.6uL	50ml	0.2mg/Kg Vinyl Chloride 2.5mg/Kg Exceptions

10.8.8.3 Using the data obtained, in labworks upload the 0.25mg/Kg MDL and manually replace the results for the exceptions, which can be viewed at Table 8.2.4.1.1, from the other (Vial 1 & Vial 3) MDL vials' data.

10.8.8.4 Uploading the MDL data this way will allow for the correct starting calibration point for each compound to be shown.

10.9 *Data Interpretation*

10.9.1 Qualitative identification is achieved based on retention time of compounds from a known standard and comparison of sample mass spectra with reference spectra generated on the instrument under the same operating condition from known standard. Qualitative identification should be achieved using the following criteria:

10.9.1.1 Selection of the peak by the data system search routine where the search is based on the peak containing specific target compound ions at a compound specific retention time.

10.9.1.2 The RRT of sample component is within ± 0.06 RRT units of the standard component, the RRT of any detect must be within ± 0.06 RRT units of the CCV run at the beginning of the 12hr

analysis shift.

10.9.1.3 The relative intensities of the characteristic ions should agree within 30% of the relative intensities of these ions in the reference spectra.

10.9.1.4 Structural isomers should be identified and reported if they have sufficiently different GC retention times.

10.9.2 For compounds not found in the calibration standards, a library search may be used to tentatively identify these compounds. The following criteria apply:

10.9.2.1 Relative intensities of major ions in the reference spectra should be present in the sample spectra.

10.9.2.2 These intensities should agree within ± 20%.

10.9.2.3 Molecular ions present in the reference spectra should be present in the sample spectra.

10.9.2.4 Ions present in the sample spectra, but not the reference spectra should be reviewed for possible background contamination of co-eluting compounds.

10.9.2.5 Ions present in the reference spectra, but not in the sample spectra should be reviewed for possible false subtraction from the sample spectra.

10.9.2.6 The Wiley library is used for tentative identification of compounds. The Wiley library is located at s:\hpchem\database\wiley275.1

10.9.2.7 Once a VOC analyte is identified, it is quantitated using the integrated abundance of the primary characteristic ion for that compound and the integrated abundance of the primary characteristic ion for the nearest internal standard to that compound. The software will calculate a concentration based on these criteria as compared to the seven point calibration curve. The quantitation of tentatively identified compounds is achieved using the formula in section 11.11.

10.9.2.8 The minimum detection limit is set at 1.0 in Chemstation, at this setting any positive result greater than 1.0 µg/ml for the target compounds will be recorded on the quantitation report. For any detects < 5ug the result is to be deleted. The result should be "lined out" and **BRL (**below reporting limit) written next to the result with date and analyst's initial.

10.9.2.9 The resulting concentration for the TIC should be reported indicating that the value is an estimate and which internal standard was used for the calculation.

10.9.3 The quantitation report software performs all of these calculations and the final concentrations are correct amounts and estimates, unless a dilution was performed on the sample prior to analysis. If a dilution was performed, multiply the quantitation report results for the compound by this dilution factor to achieve the correct concentration. Any TIC that is reported is done so by entering its name at the bottom of the compound list and entering the calculated result as a number followed by TIE.

10.10 Laboratory Information Management System (Labworks)-After the sample batch has been analyzed it is ready to be reported. The data is uploaded to the Laboratory Information Management System (LIMS). The following are the LIMS test codes for 8260:

WATER SAMPLES

$826BW	Sample results
$B_826BW	Method blanks results
$LS826BW	LCS (Laboratory Control Spike) result
$LD826BW	LCSD (Laboratory Control Spike Duplicate)
$LR826BW	LCS (Laboratory Control Spike) recovery calculation
$L2826BW	LCSD (Laboratory Control Spike Duplicate) recovery calculation

$LP826BW	Precision calculation between the LCS and the LCSD
$LA826BW	Amount spiked for LCS and LCSD
$S_826BW	MS (Matrix Spike) result
$D_826BW	MSD (Matrix Spike Duplicate) result
$R_826BW	MS (Matrix Spike) recovery calculation
$RD826BW	MSD (Matrix Spike Duplicate) recovery calculation
$P_826BW	Precision calculation between the MS and the MSD
$A_826BW	Amount spiked for Matrix Spike and Matrix Spike Duplicate
$ML826BW	MDL Result
$MA826BW	MDL Spike Amount
$INSTR-826BW	MDL Instrument Identification

SOIL SAMPLES

$826BS	Sample results
$B_826BS	Method blanks results
$LS826BS	LCS (Laboratory Control Spike) result
$LD826BS	LCSD (Laboratory Control Spike Duplicate)
$LR826BS	LCS (Laboratory Control Spike) recovery calculation
$L2826BS	LCSD (Laboratory Control Spike Duplicate) recovery calculation
$LP826BS	Precision calculation between the LCS and the LCSD
$LA826BS	Amount spiked for LCS and LCSD
$S_826BS	MS (Matrix Spike) result
$D_826BS	MSD (Matrix Spike Duplicate) result
$R_826BS	MS (Matrix Spike) recovery calculation
$RD826BS	MSD (Matrix Spike Duplicate) recovery calculation
$P_826BS	Precision calculation between the MS and the MSD
$A_826BS	Amount spiked for Matrix Spike and Matrix Spike Duplicate
$ML826BS	MDL Result
$MA826BS	MDL Spike Amount
$INSTR-826BS	MDL Instrument Identification

WASTE SAMPLES

$826BT	Sample results
$B_826BT	Method blanks results
$LS826BT	LCS (Laboratory Control Spike) result
$LD826BT	LCSD (Laboratory Control Spike Duplicate)
$LR826BT	LCS (Laboratory Control Spike) recovery calculation
$L2826BT	LCSD (Laboratory Control Spike Duplicate) recovery calculation
$LP826BT	Precision calculation between the LCS and the LCSD
$LA826BT	Amount spiked for LCS and LCSD
$S_826BT	MS (Matrix Spike) result
$D_826BT	MSD (Matrix Spike Duplicate) result
$R_826BT	MS (Matrix Spike) recovery calculation
$RD826BT	MSD (Matrix Spike Duplicate) recovery calculation
$P_826BT	Precision calculation between the MS and the MSD
$A_826BT	Amount spiked for Matrix Spike and Matrix Spike Duplicate

$ML826BT	MDL Result
$MA826BT	MDL Spike Amount
$INSTR-826BT	MDL Instrument Identification

QC SAMPLE BATCHING

#Q$826BS	QC Batch for 8260B Soil
#Q$826BT	QC Batch for 8260B Waste
#Q$826BW	QC Batch for 8260B Water

10.10.1 Samples are auto-uploaded into Labworks
From Labworks choose "Load" and a window's explorer screen will appear. Go to the "G" drive and choose the appropriate computer name to access your data. A list of all the sample .RR file names will appear. Click on this file and then "OK." and the results should be up-loaded into Labworks.

10.10.2 Verify the results by comparing to the paper hard copy printout to the screen results that were uploaded, review analyst, date of analysis, time of analysis, and all detected/non-detected results.

10.10.3 If a sample was above the range of the calibration curve and a dilution was made the reporting limits must be modified by multiplying the dilution factor (i.e. for a 2x dilution the reporting limit is multiplied by 2, so a RL of 10 will become 20 due to the dilution factor).

11 CALCULATIONS

11.1 Daily Calibration Verification and Continuing Calibration
A 50 µg/L calibration standard ensures the instrument's SPCCs and CCCs meet method performance criteria. For any 12 hour analysis period, prior to sample analysis, a one point daily continuing calibration verification is performed. The System Performance Check Compounds (SPCCs) must meet the minimum average relative response factor. For the Calibration Check Compound (CCCs) the percent drift for each CCC is not to exceed 20% of the initial calibration. If the continuing calibration does not meet method performance criteria then the instrument must be recalibrated.

Calculate the percent drift using the following equation:

Equation 11.1.1 $\%Drift = (Ci - Cc)/Ci \times 100$

where:
Ci = Calibration Check Compound standard concentration.
Cc = Measured concentration using selected quantitation method.

11.2 Relative Response Factor (*RRF*)
Relative Response Factor: Calculate the relative response factors (*RRF*) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

Equation 11.2.1 $$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

RRF = Relative response factor
A_x = Area of the primary ion for the compound to be measured
A_{is} = Area of the primary ion for the internal standard
C_{is} = Concentration of internal standard spiking mixture, µg/L
C_x = Concentration of the compound in the calibration standard, µg/L

11.3 Mean Relative Response Factor ($\overline{RRF}$)

Mean Relative Response Factor: Calculate the mean $\overline{RRF}$ for each compound by averaging the values obtained at the seven concentrations using the following equation:

where:

$\overline{RRF}$ = Mean relative response factor
x_i = RRF of the compound
n = Number of values

11.4 Percent Relative Standard Deviation (*%RSD*)

Using the RRFs from the initial calibration, calculate the *%RSD* for all target compounds using the following equations:

Equation 11.4.1 $$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} x\ 100$$

Equation 11.3.1 $$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n}$$

And where:

SD_{RRF} = Standard deviation of initial response factors (per compound)
RRF_i = Relative response factor at a concentration level
$\overline{RRF}$ = Mean of initial relative response factors (per compound).
n = Number of values

Equation 11.4.2 $$SD_{RRF} = \sqrt{\sum_{i=1}^{n} \frac{(RRF_i - \overline{RRF})^2}{n-1}}$$

11.5Relative Retention Times (*RRT*)

The retention time for each internal standard must be within □30 seconds of the retention time of the internal standard in the most recent valid calibration. Relative retention time of each analyte must be within ± 0.06 RRT units of the RRT. Calculate the *RRT*s for each target compound over the initial calibration range using the following equation:

Equation 11.5.1 $$RRT = \frac{RT_c}{RT_{IS}}$$

where:

Rt_c = Retention time of the target compound
RT_{IS} = Retention time of the internal standard.

11.6 Mean of the Relative Retention Times ($\overline{RRT}$):

Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound over the initial calibration range using the following equation:

where:

$\overline{RRT}$ = Mean relative retention time for the target compound for each initial calibration standard
RRT = Relative retention time for the target compound at each calibration level
n = Number of values

Equation 11.6.1 $$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n}$$

11.7 Tabulate the area response (Y) of the primary ion and the corresponding concentration for each compound and internal standard.

11.8 Mean Area Response ($\overline{Y}$) for Internal Standard:

Calculate the mean area response ($\overline{Y}$) for each internal standard compound over the initial calibration range using the following equation:

Equation 11.8.1 $$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where:

$\overline{Y}$ = Mean area response

Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

11.9 Mean of the Retention Times ($\overline{RT}$) For Internal Standard:
Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

Equation 11.8.1 $$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where:

$\overline{RT}$ = Mean retention time
RT = Retention time for the internal standard for each initial calibration standard.
n =Number of values

11.10 Percent Difference (%D):
Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

Equation 11.9.1 $$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} x\ 100$$

where:

RRF_c = RRF of the compound in the continuing calibration standard
$\overline{RRF}$ = Mean RRF of the compound in the most recent initial calibration.

11.11 Sample Concentration Calculation.

Equation 11.10.1 $$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

where:

C_x = Compound concentration, µg/L
A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the internal standard spiking mixture, µg/L
RRF =Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration
DF = Dilution factor. If no dilution is performed, DF = 1

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating Procedure. (See SOP reference 13.3)

13 Reference

13.1 Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), EPA SW846, Revision 2, December 1996.
13.2 GA EPD Laboratory SOPs – Initial Demonstration of Capability SOP 6-001, online revision and Continuing Demonstration of Capability SOP 6-002, online revision.
13.3 GA EPD Laboratory SOP – EPD Laboratory Waste Management SOP, SOP 6-015, online revison.
13.4 GA EPD Laboratory SOP – Determination of Method Detection Limit, SOP 6-007, online revision.
13.5 GA EPD Laboratory SOP – EPD Laboratory Procedures for Control Charting and Control Limits, SOP 6-025, online revision.
13.6 GA EPD Laboratory Quality Assurance Plan, online revision.
13.7 GA EPD Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.

14 Practical Quantitation Limits, Precision and Accuracy Criteria and Quality Control Approach

14.1 Refer to Appendix A, Table A.1 for recovery, precision, and accuracy criteria.

Table 14.2 RLs for 8260B

Parameter/ Method	Analyte	Matrix Water		Matrix Soil		Matrix Waste	
		RL	Unit	RL	Unit	RL	Unit
VOCs in 8260B	Dichlorodifluoromethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Chloromethane	10.0	µg/L	10.0	µg/kg	0.50	mg/kg
	Bromomethane	10.0	µg/L	10.0	µg/kg	0.50	mg/kg
	Vinyl chloride	2.0	µg/L	2.0	µg/kg	0.25	mg/kg
	Chloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Methylene chloride	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Trichlorofluoromethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Acetone	100.0	µg/L	100.0	µg/kg	5.00	mg/kg
	Dibromomethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	trans-1,2-Dichloroethene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Iodomethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
VOCs in 8260B	Carbon disulfide	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,1-Dichloroethene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,1-Dichloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	cis-1,2-Dichloroethene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	2,2-Dichloropropane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Bromochloromethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Chloroform	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,1-Dichloropropene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2-Dichloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	2-Butanone	100.0	µg/L	100.0	µg/kg	5.00	mg/kg
	1,1,1-Trichloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Carbon tetrachloride	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Vinyl acetate	50.0	µg/L	50.0	µg/kg	2.50	mg/kg
	Bromodichloromethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2-Dichloropropane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Trichloroethene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Benzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	cis-1,3-Dichloropropene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	trans-1,3-Dichloropropene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Dibromochloromethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,1,2-Trichloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Bromoform	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2,3-Trichloropropane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	4-Methyl-2-pentanone	50.0	µg/L	50.0	µg/kg	2.50	mg/kg
	2-Hexanone	50.0	µg/L	50.0	µg/kg	2.50	mg/kg
	Tetrachloroethene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,3-Dichloropropane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg

Table 14.2 RLs for 8260B							
Parameter/ Method	**Analyte**	**Matrix Water**		**Matrix Soil**		**Matrix Waste**	
		RL	**Unit**	**RL**	**Unit**	**RL**	**Unit**
	1,1,2,2-Tetrachloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Toluene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2-Dibromoethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Chlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Ethylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,1,1,2-Tetrachloroethane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Styrene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	p,m-Xylene	10.0	µg/L	10.0	µg/kg	0.50	mg/kg
	o-Xylene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Isopropylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Bromobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	n-Propylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	2-Chlorotoluene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,3,5-Trimethylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
VOCs in 8260B	4-Chlorotoluene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	tert-Butylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2,4-Trimethylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	sec-Butylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,3-Dichlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	p-Isopropyltoluene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,4-Dichlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	n-Butylbenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2-Dichlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2-Dibromo-3-chloropropane	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2,4-Trichlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Hexachlorobutadiene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Naphthalene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	1,2,3-Trichlorobenzene	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
	Acrylonitrile	200.0	µg/L	N/A	N/A	N/A	N/A
	trans-1,4-Dichloro-2-butene	100.0	µg/L	N/A	N/A	N/A	N/A
	Methyl tert-butyl ether	5.0	µg/L	5.0	µg/kg	0.25	mg/kg
SOW							
	1,1,2-Trichloro-1,2,2-trichloromethane	10.0	µg/L	10.0	µg/kg	0.5	mg/kg
	Methyl acetate	10.0	µg/L	10.0	µg/kg	0.5	mg/kg
	Cyclohexane	10.0	µg/L	10.0	µg/kg	0.5	mg/kg
	Methylcyclohexane	10.0	µg/L	10.0	µg/kg	0.5	mg/kg

Table 14.3 Summary of Calibration and QC Procedures for Method EPA 8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
8260B	Volatile Organics	Seven -point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF≥0.10 (≥ 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane); CCC %RSD must be ≤ 30%. All Cmpds % RSD ≤ 15%. *Option # 1* linear regression for any analytes Corr r ≥ 0.990. *Option # 2* non-linear regression Corr r ≥ 0.990.	Correct problem then repeat initial calibration.	
		Second-source calibration verification, ICV	Once per seven-point initial calibration (50µg/L concentration level)	All performance analytes within ±30% of expected value (SPCCs & CCCs)	Correct problem. Repeat another second source run. If problem persist repeat initial calibration.	
8260B	Volatile Organics	Calibration verification, CCV	Daily, before sample analysis, every12 hours of analysis time	SPCCs average RF ≥ 0.1; and CCCs ±20% drift; Compounds should have a %D of ±20%. Linear and Quadratic fit <20% deviation from nominal. It is permissible to have 5% of the compounds fail with %D > 20% (positive) if no hits for those compounds in the batch.	Rerun CCV one more time. If correct, use sample results. If not, correct problem then repeat initial calibration verification and rerun all samples since the last successful calibration verification	
		Initial Demonstration, IDC: 4 replicate analyzes of a QC sample, 1 blind sample	Once per analyst	QC acceptance criteria, Appendix A	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration, CDF: 4 replicates analyzes of a QC sample	Every 6 months	QC acceptance criteria, Appendix A	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description, valid for 12 hours.	Retune instrument and verify	
		Internal Standards	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds: EICP area within -50% and+200% of Initial Calibration from mid-point standard (50µg/L)	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Flag samples associated with failed internal standard with "J"
		Method Blank	One per analytical batch	No analytes detected >RL	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	If unable to re-analyze, flag with a "B"

Table 14.3 Summary of Calibration and QC Procedures for Method EPA 8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria Appendix A	Correct problem then reanalyze the LCS and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Matrix spike & Matrix spike dup	One MS & MSD per analytical batch	QC acceptance criteria Appendix A	Flag report	
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria Appendix A	Flag report	
8260B	Volatile Organics	MDL study	Continuous with each batch, or 7-days if method /instrument modified. Updated once per year	Detection limits established shall be less than the RLs in Table 14.2	None	
		Detect amount above the highest concentration of calibration curve	None	All detected analytes must be below curve maximum	Sample must be diluted to below highest point of curve and reanalyzed. Adjust RL as needed for dilution factor.	If unable to rerun, apply "E" to all analytes above initial calibration range

Appendix A – Quality Assurance Criteria for Method EPA 8260B*

Table A.1 - Quality Assurance Criteria for EPA 8260B

QC Type	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)	Accuracy Waste (%R)	Precision Waste (RPD)
LCS/ LCSD	1,1-Dichloroethene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Benzene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Trichloroethene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Toluene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Chlorobenzene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
MS/ MSD	1,1-Dichloroethene	**23.9-164**	<30%	**60.8-130**	<40%	**70-130****	<50%
	Benzene	**64.6-139**	<30%	**70-130**	<40%	**70-130****	<50%
	Trichloroethene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Toluene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
	Chlorobenzene	**70-130**	<30%	**70-130**	<40%	**70-130****	<50%
Surrogates	Dibromofluoromethane	**70-130**	N/A	**70-130**	N/A	**70-130****	N/A
	1,2-Dichoroethane-d4	**70-130**	N/A	**70-130**	N/A	**70-130****	N/A
	Toluene-d8	**70-130**	N/A	**70-130**	N/A	**70-130****	N/A
	Bromofluorobenzene	**70-130**	N/A	**68.2-130**	N/A	**70-130****	N/A

**LCS/LCSD MS/MSD recovery and precision limits, and surrogate recovery limits based on control charts of data collected from 1/1/2017 to 12/31/2018.*
***Default Limits, after 20 samples, limits will be re-calculated*

Updates:
Updated for online revision
Appendix A added