



APPENDIX I

Airtech Environmental Laboratories – Method 8260B SOP



1. SCOPE and APPLICATION

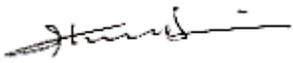
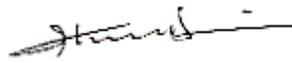
- 1.1. The intention of this SOP is to provide guidelines for the qualitative and quantitative determination of volatile compounds in water samples. This is a purge and trap gas chromatographic/mass spectrometry method applicable to the determination and quantitation of volatile organic compounds. The compounds determined by this method are insoluble or slightly soluble in water and methanol and are capable of being eluted, without derivatization, from a gas chromatograph fused silica capillary column coated with a slightly polar silicon phase.
- 1.2. Analytes highly soluble in water will have higher quantitation limits because of poor purging efficiency. 2-Chloroethylvinyl ether cannot be reported from samples containing acid preservation.

2. RANGES, SENSITIVITY, DETECTION LIMITS

- 2.1. Minimum reporting limits (PQLS) can be obtained in the absence of interferences.

3. RESPONSIBILITIES

- 3.1. This SOP is intended for the use of experienced analysts, well versed in the operation of purge and trap systems, gas chromatography and mass spectroscopy, and the interpretation of mass spectra. It should also be used for the training of technicians and chemists in the above referenced methods, and as a reference for data reviewers for data generated by use of this SOP.
- 3.2. It is the responsibility of the analyst to perform this method in accordance with the most current version of this SOP.
- 3.3. It is the responsibility of the analyst to perform this method in a manner that protects the health and safety of the analyst and fellow employees. The toxicity and carcinogenic properties of chemicals used in this method have not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Stock standard solutions of these compounds should be handled in a manner that minimizes exposure and it is recommended that solutions be prepared in a fume hood. Protective equipment, such as safety glasses, must be worn by analysts while performing this analysis. All compressed gases cylinders must be secured with chains or straps. Analysts must be trained in tank changing procedures.

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3.4. MSDS: All reagents and standards used for analysis must be accompanied by a manufacturer's Material Safety Data Sheets (MSDS) upon arrival to the lab. The MSDS are to be kept on file in the lab and made available to laboratory personnel. As a part of training process analysts must make themselves familiar with the contents of MSDS.

3.5. It is the responsibility of the Laboratory Director or designee to provide proper training and equipment required to perform this method.

3.6. Data Reviewer: The data reviewer must ensure the submitted data package is complete.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Impurities in the purge gas, organic compounds, and solvent vapors from the laboratory account for the majority of contamination problems.

4.3. Samples can be contaminated by diffusion of VOC's (particularly by fluorocarbons and methylene chloride) into the sample during shipment and storage. A trip blank prepared from reagent water and carried through the sampling and handling procedure can serve as a check on such contamination.

4.4. Glassware with soap residue or other contaminants may cause degradation of some compounds. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand rinsed very carefully to avoid this problem. Disposable glassware is used whenever possible.

4.5. Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. Whenever possible, a blank should be analyzed immediately after a high concentration sample to check for carryover. If carryover is suspected, the affected samples should be reanalyzed. The trap is subject to contamination; therefore, frequent baking out and purging of the entire system may be required.

4.6. Analytical difficulties may be encountered when analyzing for compounds with a low molecular weight, halogenated hydrocarbons, aromatic compounds, ketones, nitriles, acetates, acrylates, ethenes, and sulfides.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Aqueous samples are collected in triplicate using 40 mL screw cap vials each equipped with a PTFE-faced silicone septum. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination.

5.1.1. All aqueous samples should contain 4-5 drops of 1:1 HCl so that the sample pH remains at less than 2 to prevent decomposition of method analytes by microbial



agents. The actual pH of each water sample is determined by test strip at the time of analysis and recorded on the Daily Cover Sheet.

- 5.1.2. Aqueous samples requiring analysis for 2-chloroethyl vinyl ether must be collected in unpreserved vials.
- 5.1.3. Aqueous samples must be chilled to $4.0 \pm 2.0^{\circ}\text{C}$ on the day of collection and maintained at that temperature until analysis. Samples must be analyzed within 14 days of collection.
- 5.1.4. All samples must be collected with no air bubbles present in the vial upon sealing. A bubble smaller than the size of a pea (i.e. bubble not exceeding $\frac{1}{4}$ " or 6 mm) is permissible. Notation of headspace will be made in the case narrative of the final report.

6. EQUIPMENT

6.1. Gas Chromatograph

- 6.1.1. Gas Chromatograph: Hewlett-Packard Gas Chromatograph 5890 equipped with electronic pressure programmable split-splitless injection port.
- 6.1.2. Purge and Trap: Tekmar 2016 Purge and Trap Concentrator or equivalent with 10 mL sample handling capability.
- 6.1.3. Trap: #3 or Vocarb 3000 (K) from Supelco, or similar.
- 6.1.4. Column: 25 m x 0.20 mm ID bonded-phase silicone coated fused silica capillary column (DB624 or equivalent) with a film thickness of 1.12 micron, or similar.
- 6.1.5. Detector: Hewlett-Packard Mass Selective Detector 5972, or similar.

6.2. Volumetric Flasks: 5 mL and 10 mL Class A, with ground-glass or Teflon stoppers.

6.3. Syringes: Various sizes, gas tight.

6.4. Vials

- 6.4.1. 40 mL VOA vials, with Teflon lined septa.
- 6.4.2. 2 mL vials with Teflon lined screw caps for storing standards.
- 6.4.3. 1 mL micro-reaction vials with mininert valves for storing standards.
- 6.4.4. Scintillation vials, 20 mL with PTFE lined screw caps.
- 6.4.5. pH test strips 0-14 units



7. REAGENTS AND STANDARDS

Note: Concentrations and manufacturers/vendors of standards may vary. Adjust calculations accordingly to make standards at concentrations required for analysis.

7.1. Methanol: High Purity, Purge & Trap grade. Store at room temperature. The expiration date is the date specified by the manufacturer, or one year after receiving if no date is specified by the manufacturer.

7.2. Internal standards (IS)

7.2.1. The internal standards recommended are as follows:

- Pentafluorobenzene
- 1,4-Difluorobenzene
- Chlorobenzene-d₅
- 1,4-Dichlorobenzene-d₄

7.2.2. Stock Internal Standard (2.0 mg/mL): A certified prepared internal standard mix is purchased from Accustandard or another commercial supplier at a high concentration in methanol. Follow manufacturer's instructions for storage requirements and expiration dates. Document receipt of the standard in the "Reagent/Standard Receipt Logbook".

7.2.3. 8260 Internal Standard: From the Stock Internal Standard, prepare a Working Internal Standard solution at a concentration of 25 µg/mL. To prepare add approximately 9 mL of methanol to a 10 mL Class A volumetric flask. Add 125 µL of the Stock Internal Standard (2000ppm). Dilute to volume with methanol. Record the preparation of this solution in the "VOA Standard Prep Log" Log Book. Store the solution at < -10°C in labeled vials with Teflon lined screw caps. This solution must be replaced when the manufacturer's expiration date has passed.

7.3. Surrogate Standards

7.3.1. The recommended surrogates are as follows:

- Dibromofluoromethane
- Toluene-d₈
- 4-Bromofluorobenzene

7.3.2. Stock Surrogate Standard (2.0 mg/mL): A certified prepared surrogate mix is purchased from Accustandard or another commercial supplier, each at a high concentration in methanol. Follow manufacturer's instructions for storage requirements and expiration dates. Document receipt of this standard in the "Reagent /Standard Receipt Logbook".

7.3.3. Working Surrogate Standard Solution: From the stock surrogate standard, prepare a Working Surrogate Solution at a concentration of 25 µg/mL. To prepare add approximately 9 mL of methanol to a 10 mL Class A volumetric flask. Add 125 µL of



the Stock Surrogate Standard (2000ppm). Dilute to volume with methanol. Record the preparation of this solution in the “VOA Standard Prep Log” Log Book. Store the solution at $< -10^{\circ}\text{C}$ in labeled vials with Teflon lined screw caps. This solution must be replaced when the manufacturer’s expiration date has passed.

- 7.3.4.** GC/MS Tuning Standard - 4-Bromofluorobenzene (BFB): The surrogate-working standard that contains BFB is used for the tuning standard.

7.4. Primary Standards

- 7.4.1.** Stock Primary Standard: Certified calibration standard mixes (2000ug/mL or 5000ug/mL) are purchased from Ultra or another commercial supplier, each at a high concentration in methanol. When the mixes are combined, the resulting solution shall contain each analyte for detection by this method. All solutions prepared from these mixes shall be considered Primary Standards. Follow manufacturer’s instructions for storage requirements and expiration dates. Document the receipt of these standards in the Reagent Logbook.

- 7.4.2.** 8260 Primary Working Standard (50 $\mu\text{g}/\text{mL}$): Prepare a Working Primary Solution that will contain each target analyte at the final concentrations listed in the chart below. Prepare by combining and diluting required amounts of the stock primary mixes to a final volume of 5 mL in methanol. Record the preparation of this solution in the “VOA Standard Prep Log”. Store the solution at $< -10^{\circ}\text{C}$ in labeled vials with Teflon lined screw caps. This solution must be replaced when the manufacturer’s expiration date has passed.

- 7.4.3.** 8260 Primary Working Standard 5 $\mu\text{g}/\text{mL}$: From the 8260 Primary Standard 50 $\mu\text{g}/\text{mL}$, make a 5 $\mu\text{g}/\text{mL}$ primary standard by adding 100 μL of the 50 $\mu\text{g}/\text{mL}$ Primary standard to 900 μL Methanol.

Note: This chart is an example of manufacturers and their stock concentrations. Equivalent suppliers may be used. Concentrations may change. Volume of stock added to 10 mL is adjusted to reach the final working standard concentration.



Preparation of Primary Working Standard Solutions

50 µg/mL Working Standard:
Final Volume = 10 mL MeOH

Name of Std.	Conc. Stock Std.	Amount added to 5 mL volumetric	Final working std Concentration
Vinyl Acetate	2000 µg/mL	250 µL	50 µg/mL
24 Compounds	2000 µg/mL	250 µL	50 µg/mL
54 Compounds	2000 µg/mL	250 µL	50 µg/mL
Az Custom Mix	2000 µg/mL	250 µL	50 µg/mL
502.2 Calibration Mix	2000 µg/mL	250 µL	50 µg/mL

7.5. Calibration Standards: Standards for initial calibration of the instrument and the continuing calibration verification are prepared from the Primary.

7.5.1. Initial Calibration Standard: Prepare standards for initial calibration of the instrument at eight concentration levels.

Initial Calibration Standards for Method 8260B

Final Volume = 10.0 mL

50 µg/mL Working Std (µL)	25 µg/mL Surrogate	Surrogate Conc. µg/L	Standard Conc. µg/L
*1 µL	0.5 µL	2.5	0.5
*2 µL	1 µL	5.0	1.0
*5 µL	2 µL	10	2.5
*10 µL	4 µL	20	5.0
2 µL	10 µL	50	1.0
5 µL	14 µL	70	25
10 µL	16 µL	80	50
20 µL	20 µL	100	100
40 µL			200

* 40 µg/mL Working Std used for these points

NOTE: A minimum of five levels is required for all target compounds being analyzed. Use a minimum of six standards if a linear or higher order regression is used for quantitation. Prepare calibration standards by diluting appropriate amounts of the primary and surrogate solutions to a final volume of 100 mL with water. Each calibration standard should contain every analyte and surrogate for detection by this method. Fresh standards should be prepared each time an initial calibration is performed.



7.6. Secondary Source Standards: The secondary source standards contain all of the target compounds that are contained in the Working Primary Standards.

7.6.1. Stock Secondary Standard (2.0 mg/mL): Certified calibration standard mixes are purchased from Restek/Accustandard or another commercial supplier, each at a high concentration in methanol. Follow the manufacturer's instructions for storage requirements and expiration dates. Document receipt of the standards in the Reagent Logbook.

7.6.2. 8260 Spike Standard (50 µg/mL): Prepare a Working Secondary solution (see table below), which will contain each target analyte at the final concentrations of 50 µg/mL. Prepare by combining and diluting required amounts of the stock primary mixes to a final volume of 5 mL in methanol. Record the preparation of this solution in the "VOA Standard Prep Log". Store the solution at < -10°C in labeled vials with Teflon lined screw caps. This solution must be replaced when the manufacturer's expiration date has passed or every 3 months, whichever comes first.

8. INSTRUMENTATION

8.1. Instrument (GC/MSVOA1)

8.1.1. Purge and Trap

Purge Time	11.0 min
Dry Purge	0 min
Desorb Preheat	245 °C
Desorb	1.0 min at 250 °C
Bake	10.0 min at 260 °C

8.1.2. Gas Chromatograph

8.1.2.1. Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, and QC.

8.1.2.2. The following are the recommended GC analytical conditions, actual conditions may vary:

Initial Column Temperature; Hold	35 °C for 4 min
First Temperature Ramp	200°C at °C / 0 min
Final Temperature, Hold	8° / min to 220°C for 2.37 min
Injector Temperature	180 °C
Source Temperature	230 °C
Injector	split @ 1:50
Sample Volume	10 mL



8.1.3. Mass Spectrometer

8.1.4. The following are the required mass spectrometer analytical conditions:

Electron Energy	70 volts (nominal)
Mass Range	35 to 270 amu
Scan Time	1.8 scans per second

9. PROCEDURE

9.1. The GC/MS must be turned on a minimum of 12 hours prior to the start of analysis.

9.2. The GC/MS system must be tuned to meet BFB target tune criteria using a PFTBA tuning standard. All tune criteria must be met according to manufacturer's specifications (See Chapter 3 Tuning procedures in the MS ChemStation User's Guide).

9.3. Prior to the analysis of any calibration standards, samples, or QC, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing 4-Bromofluorobenzene (BFB).

9.4. BFB Tune Check: Prepare the Tune Check by adding 10 μ L of the 25 μ g/mL 8260 Surrogate Standard and 10 μ L of the 25 μ g/mL Internal Standard into 5 mL of Nanopure water. This will result in a surrogate concentration of 50 ppb.

9.4.1. The analysis of the instrument performance check solution must meet the ion abundance criteria.

9.4.2. The mass spectrum of BFB should be evaluated by taking the scan that occurs at the peak apex. Scans that occur within ± 3 scans from the peak apex, or an average of scans taken within that range, are also acceptable. Background subtraction must be performed. It should be straightforward and designed only to eliminate column bleed or instrument background ions.

9.4.3. The instrument performance check solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are analyzed.

Note: All subsequent standards, samples, and QC associated with a BFB analysis must use identical mass spectrometer instrument conditions.

Note: Tune checks can also be taken from CCV runs.

Note: The 12-hour period for a GC/MS system instrument check begins at the moment of injection of the BFB analysis. A sample is considered to be within this 12 hour period if the injection time is before the 12 hours end, even though the run may extend after the 12 hour period.



9.5. Initial GC/MS Calibration

9.5.1. An initial calibration curve must meet the requirements outlined in this section before any samples or QC can be analyzed. After the GC/MS system has been tuned and the instrument performance check solution has been analyzed, prepare a minimum of five levels of calibration standards, the lower one of which is at the lowest reporting limit used for each compound. A minimum of six standards must be prepared if first or higher order regressions are used for quantitation.

9.5.2. Initial Calibration Verification using %RSD: The GC/MS software will calculate and generate a report with the %RSD. Alternatively, %RSD can be calculated using the following equations.

9.5.2.1. Response Factor (RF): Tabulate the area of the primary characteristic ion against the concentration for each target and surrogate compound for each initial calibration standard. Calculate the RF for each compound relative to one of the internal standards using the following equation:

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the internal standard (µg/L)

C_x = Concentration of the compound to be measured (µg/L)

9.5.2.2. Percent Relative Standard Deviation (%RSD): Calculate the %RSD of the RF values for the initial calibration using the following equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where:

$$\text{Standard Deviation} = \sqrt{\sum_{i=1}^n \frac{(x_i - \bar{x})^2}{n-1}}$$



Where:

- x_i = each individual value used to calculate the mean
- \bar{x} = the mean of n values
- n = the total number of values

9.5.2.3. Initial calibration must meet acceptance criteria. For Method 8260B, the RSD of any target analyte must be 20% or less, with the exception of the Calibration Check Compound (CCC) (see table below) which must be equal or less than 30% (*unless a linear regression model was used for the compound*). When the response factors fall within the appropriate criteria, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation. If the % RSD for any compound is greater than 20% (30% for CCC compounds), the response factors will not be used for calibration.

Calibration Check Compounds-8260B	Maximum %RSD	Maximum %D (CCV)
1,1-Dichloroethene	30%	20%
Chloroform	30%	20%
1,2-Dichloropropane	30%	20%
Toluene	30%	20%
Ethylbenzene	30%	20%
Vinyl Chloride	30%	20%

9.5.3. Initial calibration evaluation using r or r^2 . The GC/MS software will calculate and generate a report with the r or r^2 for each initial calibration compound. In this case, a calibration curve of area ratio versus concentration using first or higher order regression is used. The r^2 must be 0.990 or greater to use the calibration curve for quantitation. A minimum of six calibration standards must be used for first or higher order regressions. Do not include or force zero for the linear or quadratic regression.

9.5.3.1. Create a linear regression curve from the known concentrations of the Calibration Blank and all the Curve Standards and their respective absorbance readings. Calculate the correlation coefficient (r value) for the curve and record it on the instrument print out. The coefficient can be calculated as follows:

$$y = mx + b$$

Where:

- y = Calibration Standard absorbance
- m = Slope
- x = Concentration of calibration standards
- b = Intercept



And:

$$m = \frac{n\sum(xy) - (\sum x)(\sum y)}{n\sum x^2 - (\sum x)^2}$$

$$b = \frac{\sum y - m\sum x}{n}$$

$$r = \frac{n\sum(xy) - \sum x\sum y}{\sqrt{[n\sum(x^2) - (\sum x)^2][n\sum(y^2) - (\sum y)^2]}}$$

9.5.3.2. A second order (quadratic) regression (see equation below) may be used; however, a minimum of six calibration points is required. The r^2 must be 0.990 or greater (or r must be 0.995 or greater) to use the calibration curve for quantitation.

$$Q = ax^2 + bx + c$$

9.5.4. A system performance check must be performed to ensure that minimum average RFs are met before the initial calibration can be used. Specific compounds have been designated as System Performance Check Compounds (SPCC) for volatile analysis and are listed in the table below along with their minimum acceptable RF. These SPCC typically have very low RFs and tend to decrease in response as the chromatographic system begins to deteriorate. If the minimum RFs are not met, the system must be evaluated and corrective action taken. Once the problem has been determined and corrective action has been taken, repeat the initial calibration.

SPCC	ICAL Minimum RF	CCV Minimum RF
Chloromethane	0.100	0.100
1,1,-Dichloroethane	0.100	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

9.5.5. Relative Retention Time (RRT): Calculate the (RRT) of each compound in each level of the initial calibration using the following equation:

$$RRT = \frac{RT_A}{RT_{IS}}$$

Where:

RT_A = Absolute retention time of compound

RT_{IS} = Absolute retention time of corresponding internal standard (See Table 3 for corresponding internal standard)



The RRT of each compound in each level of the initial calibration should agree within 0.06 relative retention time units.

9.5.6. A new initial calibration curve must be analyzed whenever corrective action has been taken which may change or affect the initial calibration criteria (i.e., ion source cleaning or repair, column removal or replacement, etc.), or if the continuing calibration acceptance criteria cannot be met.

9.6. Continuing Calibration Verification

9.6.1. The working calibration curve must be verified immediately after the curve, every 12 hours and prior to analyzing samples by running a continuing calibration standard. This standard is a working solution of the calibration standard at a concentration near the midpoint of the calibration curve (20 µg/L for example). The CCC and SPCC criteria for the continuing calibration must be met before any samples are analyzed.

9.6.2. Method 8260B requires that the RF for SPCCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-Tetrachloroethane, ≥ 0.10 for Chloromethane and 1,1-Dichloroethane and > 0.10 for Bromoform. If the minimum response factors are not met, the system is evaluated and corrective action, including recalibration is taken. Response factors for SPCC may be affected by standard mixture degradation, degradation of the trap, contamination of the front end of the column and active sites in the column or purge and trap system.

9.6.3. The CCCs are also evaluated in order to verify the validity of the initial calibration. Percent Difference (PD) is calculated using the following equation:

$$\text{Percent Difference} = \frac{R1-R2}{R1} \times 100$$

Where:

- R1 = Response Factor from a five-point calibration or true value
- R2 = Response Factor from a continuing calibration or calculated value from curve

If the %D is greater than 20% for each CCC, corrective action including recalibration must be taken.

9.6.4. The non-CCC compounds must be checked against the limits. If the concentrations do not fall within the historically set limits, the system must be evaluated and corrective action taken.

9.6.5. Internal standard responses and retention times in the QC Check Standard must be evaluated after data acquisition. If the retention time for any internal standard changes by more than 0.5 minutes from retention time of the same internal



standard in the midpoint standard of the current initial calibration, the chromatographic system must be inspected for malfunctions and corrective action taken. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (50% to 200%) from the EICP area of the same internal standard in the midpoint standard of the current initial calibration, the mass spectrometric system must be inspected for malfunction and corrective action taken. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

- 9.6.6.** A new initial calibration curve must be run whenever the continuing calibration acceptance criteria cannot be met.

9.7. Sample Analysis

- 9.7.1.** Samples may be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, and QC Check Standard requirements stated above. The same instrument conditions must be employed for the analysis of samples and QC as were used for calibration. Document analysis of all samples in the Instrument Run Log.

- 9.7.2.** Environmental samples received by SAL for analysis are recorded by the client on a chain of custody form. Each sample is given a unique laboratory ID that allows it to be tracked through all laboratory procedures. Verify ID from all samples to the chain of custody and note any discrepancies between the numbers. Any discrepancies should be discussed with Sample Receiving, the Laboratory Supervisor.

- 9.7.3.** Remove the matrix samples from refrigerated storage and allow them to come to room temperature.

9.7.4. 8260B Aqueous Samples:

- 9.7.4.1.** A suspect sample may be screened prior to analysis to determine if it will foam. Add approximately 5 mL of sample to a 20 mL scintillation vial. Shake the sample to see if it foams, if the aliquot foams, dilute the sample prior to analysis.

- 9.7.4.2.** For each set of 20 or less matrix samples include a method blank, MS/MSD (if sufficient sample is available) and a LCS/LCSD.

- 9.7.4.3.** Method Blank (MB): To prepare add 10 µL of 25 ppm Internal Standard, 10 µL of 25 ppm Surrogate Standard and to 5 mL Nanopure water.

- 9.7.4.4.** Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD): Prepare 2 spikes at 25 µg/L each by adding 10 µL of Internal Standard, 10 µL of Surrogate Standard, 5 µL of the 50 µg/mL 8260 Secondary Standard to 10 mL water.



9.7.4.5. Sample Analysis: To prepare add 10 μL of 25 ppm Internal Standard, 10 μL of 25 ppm Surrogate Standard and to 10 mL of the sample aliquot.

Note: A MS/MSD must be analyzed on a 10% frequency. Precision and accuracy, either MS/MSD or LCS/LCSD pairs, must be analyzed every 12 hour shift.

9.7.4.5.1. Method Blank (MB): To prepare add 10 μL of 25 ppm Internal Standard, 100 μL of Sample Extract to 10 mL water.

9.8. Tekmar Purge and Trap Concentrator/ALS2016 Set-Up.

9.8.1. From the hand held programmer, select “schedule”, then choose “edit schedule”(E). A schedule is set up to start and stop at specified spargers. Choose the number of the spargers to be run using the numbers on the keypad and select “Enter”. This process can be repeated throughout the run to accommodate the need to add samples during the day. Be sure to choose the correct method for the concentrator.

9.8.2. After programming the schedule, select “schedule” and then select “commands” (C). Choose “Run Schedule”. The auto sampler will cycle to the first position programmed. Push start to begin the sequence.

9.9. Inject the Sample into the appropriate sparger.

9.9.1. The samples are typically analyzed in the following order: MB, LCS/LCSD, and samples including matrix samples (if enough sample available).

9.9.2. Instrument blanks should run after samples and standards containing high concentrations of target analytes or whenever there is a potential for carry over.

9.9.3. Analyze the samples using the specified instrument parameters.

9.9.4. Enviroquant Analytical Sequence Set Up.

9.9.4.1. Choose the “Sequence” menu on the HP-Chemstation. Click on “Load” and choose any previous run sequence.

9.9.4.2. When the sequence has loaded, click on “Sequence” from the menu bar, then click on “Edit Sample Log Table”.

9.9.4.3. Name each sample to be analyzed. The file naming format is MM_DD_YY_File#, i.e. 102607001.

9.9.4.4. In the “Miscellaneous” section enter the standardized sample type. These include CCV, LCS, LCSD, MS, MSD, MLBK, and SAMP.

9.9.4.5. Enter the name of the current method in the “Method” section. The method name is based on the date the current curve was analyzed.



- 9.9.4.6. Select “Repeat” to enter the appropriate information for each sample to be analyzed.
- 9.9.4.7. When the table is completed select “Enter”.
- 9.9.4.8. The “Save Sequence” screen will appear. Name the sequence using the format Instrument #_MM_DD_YY, i.e. 102607. Select “OK”.
- 9.9.4.9. Select “Sequence”, then “Position and Run”. Select the autosampler vial position that will start the run. Select “OK”.
- 9.9.4.10. A screen will appear with run options. This will normally remain unchanged. Ensure that the “Method Section to Run” is set to “Full Method”. Enter the file name.
- 9.9.4.11. Select “Run Sequence”. A screen will appear with “Procession Key Words Before Starting Sequence”. Ensure that it is set to “No”.
- 9.9.4.12. After water samples have been analyzed, measure the pH using pH paper (use the same vial that was used in the analysis). If the pH is > 2, test a second VOA, If this pH is also > 2 the Laboratory Supervisor is notified and a note is made on the “Daily Coversheet” and on all reports.

9.10. Qualitative Analysis

- 9.10.1. The target compounds shall be identified by comparison of the sample mass spectra to a reference mass spectra. Reference spectra are obtained from the 20 ppb level of the initial calibration. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. The criteria for compound identification are listed in the following sections.
- 9.10.2. For establishing correspondence of the GC retention time (RT), the sample component RT must fall within ± 30 seconds of the CCV component RT. For samples analyzed during the same 24-hour time period as the initial calibration standards, compare the sample retention times to those from the standard that is the same concentration as the QC Check Standard. If coelution of interfering components prohibits accurate assignment of the sample component RT from the total ion chromatogram, the RT should be assigned by using extracted ion current profiles for ions unique to the component of interest.
- 9.10.3. The requirements for qualitative verification by comparison of mass spectra are as follows:
 - 9.10.3.1. The relative intensities of the characteristic ions (**Table 5**) must agree within $\pm 20\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the



corresponding sample ion abundance must be between 30 and 70 percent.).

- 9.10.3.2.** Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison (i.e., ions contributing to the spectrum are interferences from coelution of another compound).
- 9.10.3.3.** Structural isomers that produce very similar mass spectra shall be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers shall be identified as isomeric pairs.
- 9.10.3.4.** If a compound does not meet this criteria, it may still be reported at the analyst's discretion. An explanation and any supporting spectral information should accompany the data package.
- 9.10.4.** A library search shall be executed for non-target sample components for the purpose of tentative identification if required by the client. For this purpose, an NIST/EPA/MSDC mass spectral library shall be used. The guidelines for making tentative identification are listed below.
 - 9.10.4.1.** Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
 - 9.10.4.2.** The relative intensities of the major ions should agree within $\pm 20\%$. (Example: for an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent).
 - 9.10.4.3.** Molecular ions present in the reference spectrum should be present in the sample spectrum.
 - 9.10.4.4.** Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds, both of which may be subtracted out. However, care should be taken not to subtract out relevant ions that occur in the peak of interest.
 - 9.10.4.5.** If, in the technical judgment of the analyst, no valid tentative identification can be made, the compound should be reported as unknown. Classification of the unknown compound (i.e. unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound) should be given, if possible.



9.10.5. For Aqueous samples, the spectra for all reported compounds and for all false positives should be printed and included in the data package.

9.11. Quantitation

9.11.1. Target components identified shall be quantified by the internal standard (IS) method. The IS used shall be the one closest to the retention time to that of a given analyte (**Table 3**). The areas of characteristic ions listed in (**Table 5**) are used for quantitation.

9.11.1.1. The area and retention times of the IS must be monitored and evaluated for all samples and QC. If the area for any IS changes by more than a factor of two (50% to 200%) from the same IS in the midpoint standard of the current initial calibration, the mass spectrometric system must be inspected for malfunction and corrective action taken. If the retention time for any internal standard changes by more than 0.5 minutes from retention time of the same internal standard in the midpoint standard of the current initial calibration, the chromatographic system must be inspected for malfunctions and corrective action taken. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required.

9.11.1.2. The samples or standards with areas outside the limits must be reanalyzed. If after reanalysis, the areas for all IS are inside the limits (50% to 200%), report the data from the reanalysis. The initial analysis may be noted on the report, but should not be reported in the result section.

9.11.1.3. If the reanalysis of the sample does not solve the problem, i.e., the areas are outside the control limits for both analyses, then submit the sample data from the first analysis. Note the results of the second analysis in the case narrative.

9.11.2. The \overline{RRF} from the initial calibration are used to calculate the concentration in the sample for the compounds that had % RSD greater than 15% in the initial calibration. If secondary ion quantification is performed, reasons for doing so must be documented. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.

9.11.3. For the compounds, which are being calibrated using first order or higher calibration curves, the data system will calculate a concentration using the equation, generated to describe the curve.

9.11.4. An estimated concentration for noncalibrated compounds, such as Tentatively Identified Compounds (TIC's), shall be made if requested by the client. The areas A_X and A_{IS} shall be from the total ion chromatograms. Use the nearest internal standard free of interferences. The \overline{RRF} for the compound shall be assumed to be 1.0.



- 9.11.5.** Manual Integration: Manual integration must be performed by an analyst proficient in the interpretation of peak resolution. Integration must be performed in accordance with the peak integration as it appears in the calibration curve. Reference SOP QAD-006.02 - Manual Integrations for detailed procedures.
- 9.11.6.** If the concentration of any compound in a sample exceeds the initial calibration range, that sample must be diluted and the sample reanalyzed. Guidelines for performing dilutions are given below.
- 9.11.6.1.** Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
- 9.11.6.2.** The dilution factor chosen should keep the responses of the largest peak for a target compound in the upper half of the initial calibration range of the instrument. However, if there are nontarget compounds that may cause harm to the GC column or MS, or if the sample appears to "foam" when being purged, a larger dilution may be made.

10. CALCULATIONS

- 10.1.** Calculate the concentration of the target compound in the sample using the average response factor (RRF) as determined previously and the following equation:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x) (I_s) (D_i)}{(A_{iS}) (RRF_{av}) (V_o)}$$

Where:

- A_x = Area of the characteristic ion for the compound to be measured
 I_s = Amount of internal standard injected in nanograms
 A_{iS} = Area of the characteristic ion for the internal standard
 RRF_{av} = Mean relative response factor for compound being measured
 V_o = Volume of water purged (mL), taking into consideration any dilutions made
 D_i = Dilution factor used (for 1:10 dilution, $D_i = 10$)

- 10.2.** Dilutions: The final concentration of any sample requiring dilution can be calculated using the following equation:

$$\text{Concentration } (\mu\text{g/L}) = C_p * D_i$$



Where:

C_P = Concentration

D_i = Dilution factor (for 1:10 dilution, D_i = 10)

10.3. Calculate percent recovery using the following equation:

10.4. Use equation below to calculate the RPD.

$$RPD = \frac{S-SD}{AVE} \times 100$$

Where:

S = Spike result

SD = Spike duplicate result

AVE = Average of S and SD

10.5. Standard deviation: Use Excel® program, *hp*® calculator or equivalent to determine the standard deviation. Alternatively, the standard deviation can be calculated using the following formula:

$$\text{Standard Deviation} = \sqrt{\frac{\sum (X - M)^2}{N - 1}}$$

Where:

∑ = The sum of all results

X = Individual results

M = The mean of all results

N = The number of samples

10.6. Calculate the relative standard deviation (RSD) using the equation below.

$$RSD = \frac{S}{X} \times 100$$

Where:

S = Standard deviation

X = Average results



11. MAINTENANCE

11.1. All maintenance done on the GC/MS will be documented in the Maintenance Logbook. The following maintenance procedures are done routinely on the GC/MS. Some procedures may be done more often than scheduled, if there is a problem in instrument performance.

11.1.1. Clean the MSD source as needed when indicated by instrument performance.

11.1.2. Replace the trap as needed when indicated by instrument performance.

11.1.3. Clean auto sampler syringe and replace o-ring as needed when indicated by instrument performance.

11.1.4. Change vacuum pump oil every year.

11.2. All maintenance performed on the instrument by a service company is documented in the Maintenance Logbook.

12. QUALITY CONTROL

12.1. Initial Calibration: An initial calibration must be evaluated using the criteria listed above. A new initial calibration curve must be analyzed after major instrument maintenance or if the CCV acceptance criteria cannot be met.

12.2. Continuing Calibration Verification: The curve must be verified after initial calibration prior to the analysis of samples and every 12 hours prior to the analysis of samples by running a CCV. The recovery of each reported compound must meet the acceptance limits. If recoveries do not meet acceptance criteria the CCV must be reanalyzed. If reanalysis confirms the original results the source of the error must be identified and corrected before samples are analyzed. All CCC compounds must meet criteria. The acceptance limits for non CCC compounds are either $100 \pm 20\%$ or determined from historical laboratory limits.

12.3. Method Blank: A method blank (MB) is analyzed in each batch of twenty or fewer matrix samples; at a minimum of once every 12 hour clock.

12.3.1. An MB is considered acceptable if no target analytes are detected at or above the method reporting limit. If the MB exceeds these criteria, the analyst must consider the analytical system to be contaminated. The contamination must be corrected and, if possible, the MB and associated samples should be reanalyzed. If reanalysis is not possible the data must be flagged accordingly.

12.4. Matrix Spike/Matrix Spike Duplicate(MS/MSD): If sufficient sample is given, a MS/MSD is analyzed for each batch of 20 matrix samples or less.

12.4.1. The recovery limits for the MS/MSD are either $100 \pm 40\%$ or derived from historical data.



12.4.2. The relative percent difference (RPD) limits are either $\leq 30\%$ or determined from historical data.

12.5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD): A LCS/LCSD is analyzed for each batch of 20 matrix samples or less.

12.5.1. The recovery limits for the LCS/LCSD are either $100 \pm 30\%$ or derived from historical data.

12.5.2. The relative percent difference (RPD) limits are either $\leq 30\%$ or determined from historical data.

12.6. Surrogates

12.6.1. The excel spreadsheet labeled “Custom Reports” will calculate the concentration of the surrogate.

12.6.2. Surrogate recovery limits are established from historical data using at least 20 analyses, and are updated at least every six months. If no historical data available, default limits will be 100 ± 30 .

12.7. Corrective Action

12.7.1. Corrective action process is initiated when data quality problems are observed or suspected. These cases include spike recovery falling out of the acceptability limits, lack of stability during calibration process, contamination of laboratory blanks, carryover, exceeded sample holding time, etc. All data quality problems should be written on all pages of the report, and the laboratory supervisor notified.

12.7.2. If the surrogate, internal standard or spiked target compound recovery results do not meet criteria, check to be sure that there are no errors in calculations, solutions, or internal standards. Also check instrument performance. If none of these items reveal a problem, corrective action must be taken and the sample or standard must be reanalyzed.

12.7.3. If the reanalysis of the sample or standard yields recoveries that are within limits, report the reanalysis.

12.7.3.1. If the reanalysis of the sample or standard does not yield recoveries within QC limits, then the problem was out of the laboratory's control. Therefore, submit data from the initial analysis and fill out an ‘Out of Control Event’ form.

12.7.3.2. If the surrogate recoveries of the sample associated with MS/MSD are not within the established control limits, it should be reanalyzed only if the MS/MSD recoveries are acceptable. If the sample and associated MS/MSD show the same pattern (i.e., recoveries outside control limits), then neither the sample, MS, nor MSD require reanalysis. Fill out and ‘Out of Control Event’ form.



12.7.3.3. If any target compound is outside of the criteria in the MS/MSD, it must be flagged on all reports with samples associated with the spiked pair. Fill out and 'Out of Control Event' form.

13. REFERENCES

- 13.1.** Tekmar 2016/2032 Auto sampler Manual.
- 13.2.** Tekmar 3100 Purge and Trap Concentrator Manual.
- 13.3.** Agilent 6890 N GC, and 5975 MS Gas Chromatograph Manual.
- 13.4.** "*Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*", Method 8260B, USEPA, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, with integrated updates I, II, IIA, IIB, and III, June 1997
- 13.5.** "*Purge and Trap for Aqueous Samples*", Method 5030B, USEPA, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, with integrated updates I, II, IIA, IIB, and III, June 1997
- 13.6.** *Determinative Chromatographic Separations*, Method 8000C, USEPA, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, with integrated updates I, II, IIA, IIB, and III, June 1997.
- 13.7.** ADHS administrative codes Title 9, Chapter 14 (a.k.a. Arizona Rules).



Table 1
PQLS for EPA Method 8260B

Compound	PQL (µg/L) Water
Acetone	20
Benzene	1
Bromobenzene	1
Bromochloromethane	1
Bromodichloromethane	2
Bromoform	5
Bromomethane	5
1,3-Butadiene	1
2-Butanone	5
n-Butylbenzene	1
Sec-Butylbenzene	1
Tert-Butylbenzene	1
Carbon Disulfide	5
Carbon tetrachloride	1
Chlorobenzene	1
Chloroethane	1
Chloroform	2
Chloromethane	5
2-Chlorotoluene	1
4-Chlorotoluene	1
Cyclohexane	1
Dibromochloromethane	1
1,2-Dibromo-3-Chloropropane	5
1,2-Dibromoethane	1
Dibromomethane	1
1,2-Dichlorobenzene	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
Dichlorodifluoromethane	1
1,1-Dichloroethane	1
1,2-Dichloroethane	1
1,1-Dichloroethene	1
Cis-1,2-Dichloroethene	1
Trans-1,2-Dichloroethene	1
1,2-Dichloropropane	1
1,3-Dichloropropane	1
2,2-Dichloropropane	2
1,1-Dichloropropene	1
Cis-1,3-Dichloropropene	1
Trans-1,3-Dichloropropene	1
Dicyclopentadiene	1
Ethylbenzene	1
Heptane	1
Hexachlorobutadiene	2
Hexane	1
2-Hexanone	5
Iodomethane	5
4-Isopropyltoluene	1
Isopropylbenzene	1



**Table 1
PQLS for EPA Method 8260B
(Cont)**

Compound	PQL (µg/L) Water
Methyl cyclohexane	1
Methylene Chloride	2
Methyl tert butyl ether	1
4-Methyl-2-pentanone	5
Naphthalene	5
Nonane	1
n-Propylbenzene	1
Styrene	1
1,1,2,2-Tetrachloroethane	1
1,1,1,2-Tetrachloroethane	1
Toluene	1
1,2,3-Trichlorobenzene	5
1,2,4-Trichlorobenzene	5
Trichloroethene	1
1,1,1-Trichloroethane	1
1,1,2-Trichloroethane	1
Tetrachloroethene	1
Trichlorofluoromethane	1
1,2,3-Trichloropropane	2
1,2,4-Trimethylbenzene	1
1,3,5-Trimethylbenzene	1
2,2,4-Trimethyl Pentane	1
Vinyl Acetate	2
Vinyl Chloride	2
m,p-Xylene	2
o-Xylene	1
Total Xylenes	3



Table 2

BFB KEY IONS AND ION ABUNDANCE CRITERIA FOR QUADRUPOLE MASS SPECTROMETERS

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Note: Alternate tuning criteria may be used (e.g. CLP, Method 524.2 or manufacturers' instructions), provided that method performance is not adversely affected.



TABLE 3

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

Pentafluorobenzene	1,4-Difluorobenzene	Chlorobenzene-d ₅	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Trichloroethylene	Chlorobenzene	Bromobezene
Chloromethane	Methyl Cyclohexane	1,1,1,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane
Vinyl chloride	1,2-Dichloropropane	Ethylbenzene	1,2,3-Trichloropropane
1,3-Butadiene	Dibromomethane	m,p-Xylene	n-Propylbenzene
Bromomethane	Bromodichloromethane	Nonane	2-Chlorotoluene
Chloroethane	2-Chloroethylvinyl ether	o-Xylene	4-Chlorotoluene
Trichlorofluoromethane	cis-1,3-Dichloropropene	Styrene	1,3,5-Trimethylbenzene
1,1-Dichloroethene	4-Methyl-2-pentanone	Bromoform	tert-Butylbenzene
Acetone	Toluene	Isopropylbenzene	1,2,4-Trimethylbenzene
Iodomethane	trans-1,3-Dichloropropene	*Toluene-d8	sec-Butylbenzene
Carbon Disulfide	1,1,2-Trichloroethane		1,3-Dichlorobenzene
Methylene Chloride	Tetrachloroethene		4-Isopropyltoluene
trans-1,2-Dichloroethene	1,3-Dichloropropane		1,4-Dichlorobenzene
Methyl tert-butyl ether	2-Hexanone		Dicyclopentadiene
Hexane	Dibromochloromethane		1,2-Dichlorobenzene
1,1-Dichloroethane	1,2-Dibromoethane		n-Butylbenzene
Vinyl Acetate	*1,2-Dichloroethane-d4		1,2-Dibromo-3-chloropropane
2,2-Dichloropropane			1,2,4-Trichlorobenzene
cis-1,2-Dichloroethene			Hexachlorobutadiene
2-Butanone			Naphthalene
Bromochloromethane			1,2,3-Trichlorobenzene
Chloroform			*4-Bromofluorobenzene
1,1,1-Trichloroethane			
Cyclohexane			
Carbon Tetrachloride			
1,1-Dichloropropene			
Benzene			
1,2-Dichloroethane			
2,2,4-Trimethyl Pentane			
Heptane			
*Pentafluorobenzene			

*Surrogate



TABLE 4

CHARACTERISTIC IONS FOR VOLATILE INTERNAL STANDARDS

INTERNAL STANDARDS	PRIMARY ION	SECONDARY IONS
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d ₅	117	82*, 119
Pentafluorobenzene	168	-
1,4-Dichlorobenzene-d ₄	152	115, 150

*The secondary ion 82 is used to quantitate due to interference of the primary ion 117.



**TABLE 5
CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS AND SURROGATES**

Compound	Primary Ion	Secondary Ion(s)
Pentafluorobenzene	168	99,137
Dichlorodifluoromethane	85	87, 50
Chloromethane	50	52, 33
Vinyl Chloride	62	64, 44
1,3-Butadiene	39	54, 28
Bromomethane	94	96,93
Chloroethane	64	66,49
Trichlorofluoromethane	101	103,66
1,1-Dichloroethene	61	96,98
Acetone	58	43
Iodomethane	142	127
Carbon Disulfide	76	44
Methylene Chloride	49	84,86,51
trans-1,2-Dichloroethene	61	96,98
Methyl tert-butyl ether	73	43,41
Hexane	57	41,42
1,1-Dichloroethane	63	65,83
Vinyl acetate	43	86
2,2-Dichloropropane	77	41,39
cis-1,2-Dichloroethene	61	96,98
2-Butanone	43	72
Bromochloromethane	49	128
Chloroform	83	85,87
1,1,1-Trichloroethane	97	99
Dibromofluoroethane	111	113/192
Cyclohexane	56	84,69
Carbon Tetrachloride	117	119,121
1,1-Dichloropropene	75	39,110
1,2-Dichloroethane-d4	65	67,51
Benzene	78	52,77
1,2-Dichloroethane	62	64,98
2,2,4-Trimethyl Penatne	57	56,29
Heptane	43	71,56
1,4-Difluorobenzene	114	63,88
Trichloroethene	130	97,132
Methyl Cyclohexane	83	55,42
1,2-Dichloropropane	63	62,76
Dibromomethane	174	93,172
Bromodichloromethane	83	85,129
2-Chloroethylvinylether	63	43



TABLE 5 (continued)
CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS AND SURROGATES

Compound	Primary Ion	Secondary Ion(s)
cis-1,3-Dichloropropene	75	77,39
4-Methyl-2-pentanone	43	68,86,100
Toluene-d8	98	70,100
Toluene	91	92,65
trans-1,3-Dichloropropene	75	77,39
1,1,2-Trichloroethane	97	83,99
Tetrachloroethene	164	129,131,166
1,3-Dichloropropane	76	41,39
2-Hexanone	43	58,100
Dibromochloromethane	129	127,131,79
1,2-Dibromoethane	107	109,81
Chlorobenzene-d5	117	82,119
Chlorobenzene	112	114,77
1,1,1,2-Tetrachloroethane	131	135,95,133
Ethylbenzene	91	106
m,p-Xylene	91	106
Nonane	43	57,71
o-Xylene	91	106
Styrene	104	78,103
Bromoform	173	171,175,255
Isopropylbenzene	105	120,77
4-Bromofluorobenzene	95	176
1,4-Dichlorobenzene-d4	150	152,115
Bromobenzene	77	156,158
1,1,2,2-Tetrachloroethane	83	85,131,133
1,2,3-Trichloropropane	75	77,110
n-Propylbenzene	91	120,92
2-Chlorotoluene	91	126,90
4-Chlorotoluene	91	126,63
1,3,5-Trimethylbenzene	105	120,77
tert-Butylbenzene	119	91,134
1,2,4-Trimethylbenzene	105	120,46
sec-Butylbenzene	105	134,19
1,3-Dichlorobenzene	146	148,111
4-Isopropylbenzene	119	146,91
1,4-Dichlorobenzene-d4	152	115,150
1,4-Dichlorobenzene	146	148,111
Dicyclopentadiene	66	39,67
1,2-Dichlorobenzene	146	148,111
n-Butylbenzene	91	92,134
1,2-Dibromo-3-chloropropane	41	39,155
1,2,4-Trichlorobenzene	179	182,97
Hexachlorobutadiene	224	227,223
Napthalene	128	127,129
1,2,3-Trichlorobenzene	180	182,145

