1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

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<th>Compound</th>
<th>CAS No.</th>
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(continued)
### Appropriate Preparation Technique

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|-----------------------------------------|---------|-------|------|------|------|------|------|------------------
| Nitrobenzene                            | 98-95-3 | c     | nd   | nd   | nd   | nd   | c    |                  
| 2-Nitropropane                          | 79-46-9 | c     | nd   | nd   | nd   | nd   | c    |                  
| N-Nitroso-di-n-butylamine               | 924-16-3| pp    | c    | nd   | nd   | nd   | c    |                  
| Paraldehyde                             | 123-63-7| pp    | c    | nd   | nd   | nd   | c    |                  
| Pentachloroethane                       | 76-01-7 | l     | nd   | nd   | nd   | nd   | c    |                  
| 2-Pentanone                             | 107-87-9| pp    | c    | nd   | nd   | nd   | c    |                  
| 2-Picoline                              | 109-06-8| pp    | c    | nd   | nd   | nd   | c    |                  
| 1-Propanol                              | 71-23-8 | pp    | c    | nd   | nd   | nd   | c    |                  
| 2-Propanol                              | 67-63-0 | pp    | c    | nd   | nd   | nd   | c    |                  
| Propargyl alcohol                       | 107-19-7| L     | nd   | nd   | nd   | nd   | c    |                  
| β-Propiolactone                         | 57-57-8 | pp    | nd   | nd   | nd   | nd   | c    |                  
| Propionitrile (ethyl cyanide)           | 107-12-0| ht    | c    | nd   | nd   | nd   | pc   |                  
| n-Propylamine                           | 107-10-8| c     | nd   | nd   | nd   | nd   | c    |                  
| Pyridine                                | 110-86-1| l     | c    | nd   | nd   | nd   | c    |                  
| Styrene                                 | 100-42-5| c     | nd   | c    | c    | c    | c    |                  
| 1,1,1,2-Tetrachloroethane               | 630-20-6| c     | nd   | nd   | c    | c    | c    |                  
| 1,1,2,2-Tetrachloroethane               | 79-34-5 | c     | nd   | c    | c    | c    | c    |                  
| Tetrachloroethene                       | 127-18-4| c     | nd   | c    | c    | c    | c    |                  
| Toluene                                 | 108-88-3| c     | nd   | c    | c    | c    | c    |                  
| Toluene-d₆ (surr)                       | 2037-26-5| c   | nd   | c    | c    | c    | c    |                  
| α-Toluidine                             | 95-53-4 | pp    | c    | nd   | nd   | nd   | c    |                  
| 1,2,4-Trichlorobenzene                  | 120-82-1| c     | nd   | nd   | c    | nd   | c    |                  
| 1,1,1-Trichloroethane                   | 71-55-6 | c     | nd   | c    | c    | c    | c    |                  
| 1,1,2-Trichloroethane                   | 79-00-5 | c     | nd   | c    | c    | c    | c    |                  
| Trichloroethene                         | 79-01-6 | c     | nd   | c    | c    | c    | c    |                  
| Trichlorofluoromethane                  | 75-69-4 | c     | nd   | c    | c    | c    | c    |                  
| 1,2,3-Trichloropropane                  | 96-18-4 | c     | nd   | c    | c    | c    | c    |                  
| Vinyl acetate                           | 108-05-4| c     | nd   | c    | nd   | nd   | c    |                  
| Vinyl chloride                          | 75-01-4 | c     | nd   | c    | c    | c    | c    |                  
| o-Xylene                                | 95-47-6 | c     | nd   | c    | c    | c    | c    |                  
| m-Xylene                                | 108-38-3| c     | nd   | c    | c    | c    | c    |                  
| p-Xylene                                | 106-42-3| c     | nd   | c    | c    | c    | c    |                  

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a See Sec. 1.2 for other appropriate sample preparation techniques
b Chemical Abstract Service Registry Number
c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
nd = Not determined
l = Inappropriate technique for this analyte
pc = Poor chromatographic behavior
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard
1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene 1,3-Dichloropropene
n-Butylbenzene 2,2-Dichloropropane
sec-Butylbenzene 1,1-Dichloropropene
tert-Butylbenzene p-Isopropyltoluene
Chloroacetonitrile Methyl acrylate
1-Chlorobutane Methyl-t-butyl ether
1-Chlorohexane Pentfluorobenzene
2-Chlorotoluene n-Propylbenzene
4-Chlorotoluene 1,2,3-Trichlorobenzene
Dibromofluoromethane 1,2,4-Trimethylbenzene
cis-1,2-Dichloroethene 1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrupole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.
2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocussed on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).
3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.
4.2 Purge-and-trap device for solid samples - Described in Method 5035.
4.3 Automated static headspace device for solid samples - Described in Method 5021.
4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.
4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.
4.6 Desorption device for air trapping media for air samples - Described in Method 5041.
4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.
4.8 Injec tion port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt 502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt 5, SPB-5, or equivalent), 1-µm film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8-µm film thickness, or equivalent.
4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.
4.15 Vials - 2-mL, for GC autosampler.

4.16 Disposable pipets - Pasteur.

4.17 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.18 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH₂OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, H(OCH₂CH₂),nOH - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to
5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.
5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

**NOTE:** Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

<table>
<thead>
<tr>
<th>Gas Volume</th>
<th>Calibration Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 µL</td>
<td>20 µg/L</td>
</tr>
<tr>
<td>100 µL</td>
<td>50 µg/L</td>
</tr>
<tr>
<td>200 µL</td>
<td>100 µg/L</td>
</tr>
<tr>
<td>300 µL</td>
<td>150 µg/L</td>
</tr>
<tr>
<td>400 µL</td>
<td>200 µg/L</td>
</tr>
</tbody>
</table>
5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

<table>
<thead>
<tr>
<th>Gas Volume</th>
<th>Calibration Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 µL</td>
<td>1 µg/L</td>
</tr>
<tr>
<td>20 µL</td>
<td>2 µg/L</td>
</tr>
<tr>
<td>50 µL</td>
<td>5 µg/L</td>
</tr>
<tr>
<td>100 µL</td>
<td>10 µg/L</td>
</tr>
<tr>
<td>250 µL</td>
<td>25 µg/L</td>
</tr>
</tbody>
</table>

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d₈, 4-bromofluorobenzene, 1,2-dichloroethane-d₄, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d₅, and 1,4-dichlorobenzene-d₄. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards - There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.
5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.
7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

7.2 Recommended chromatographic conditions

7.2.1 General conditions

- Injector temperature: 200 - 225°C
- Transfer line temperature: 250 - 300°C
7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

- **Carrier gas (He) flow rate:** 15 mL/min
- **Initial temperature:** 10°C, hold for 5 minutes
- **Temperature program:** 6°C/min to 70°C, then 15°C/min to 145°C
- **Final temperature:** 145°C, hold until all expected compounds have eluted.

7.2.5 Direct injection - Column 2

- **Carrier gas (He) flow rate:** 4 mL/min
- **Column:** J&W DB-624, 70m x 0.53 mm
- **Initial temperature:** 40°C, hold for 3 minutes
- **Temperature program:** 8°C/min
- **Final temperature:** 260°C, hold until all expected compounds have eluted.
- **Column Bake out:** 75 minutes
- **Injector temperature:** 200-225°C
- **Transfer line temperature:** 250-300°C

7.2.6 Direct split interface - Column 4

- **Carrier gas (He) flow rate:** 1.5 mL/min
- **Initial temperature:** 35°C, hold for 2 minutes
- **Temperature program:** 4°C/min to 50°C, then 10°C/min to 220°C
- **Final temperature:** 220°C, hold until all expected compounds have eluted
- **Split ratio:** 100:1
- **Injector temperature:** 125°C

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

- **Mass range:** 35 - 260 amu
- **Scan time:** 0.6 - 2 sec/scan
- **Source temperature:** According to manufacturer’s specifications
- **Ion trap only:** Set axial modulation, manifold temperature, and emission current to manufacturer’s recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2-µL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of
BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

**NOTE:** All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).
The RF is calculated as follows:
\[ RF = \frac{A_s \times C_s}{A_{is} \times C_{is}} \]

where:
- \( A_s \) = Peak area (or height) of the analyte or surrogate.
- \( A_{is} \) = Peak area (or height) of the internal standard.
- \( C_s \) = Concentration of the analyte or surrogate.
- \( C_{is} \) = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

- Chloromethane 0.10
- 1,1-Dichloroethane 0.10
- Bromoform 0.10
- Chlorobenzene 0.30
- 1,1,2,2-Tetrachloroethane 0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:
where:

\[ \text{RF}_i = \text{RF for each of the calibration standards} \]
\[ \text{RF} = \text{mean RF for each compound from the initial calibration} \]
\[ n = \text{Number of calibration standards, e.g., 5} \]

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene  Toluene
Chloroform  Ethylbenzene
1,2-Dichloropropane  Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

**NOTE:** Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.
NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater
than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.
7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.
If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10 µL of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

**NOTE:** It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.
7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. 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. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should 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 are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library
searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

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.

2. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

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.

5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas \( A_s \) and \( A_x \) should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.
8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.
8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each
sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.
9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

10.0 REFERENCES


7. Non Cryogenic Temperatures Program and Chromatogram, Private Communications; M. Stephenson and F. Allen, EPA Region IV Laboratory, Athens, GA.


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<tr>
<th>Compound</th>
<th>Retention Time (minutes)</th>
<th>MDL (µg/L)</th>
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$^a$ Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

$^b$ Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

$^c$ Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10°C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

$^d$ MDL based on a 25-mL sample volume.
### TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

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<th>Retention Time (minutes)</th>
<th>MDL $^b$ (µg/L)</th>
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<td>0.11</td>
</tr>
<tr>
<td>2-Chlorotoluene</td>
<td>16.74</td>
<td>0.08</td>
</tr>
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<td>n-Propylbenzene</td>
<td>16.82</td>
<td>0.10</td>
</tr>
<tr>
<td>4-Chlorotoluene</td>
<td>16.82</td>
<td>0.06</td>
</tr>
</tbody>
</table>
TABLE 2 (cont.)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Retention Time (minutes)</th>
<th>MDL&lt;sup&gt;b&lt;/sup&gt; (µg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Column 3&lt;sup&gt;a&lt;/sup&gt;</td>
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</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
<td>16.99</td>
<td>0.06</td>
</tr>
<tr>
<td>tert-Butylbenzene</td>
<td>17.31</td>
<td>0.33</td>
</tr>
<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>17.31</td>
<td>0.09</td>
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<tr>
<td>sec-Butylbenzene</td>
<td>17.47</td>
<td>0.12</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>17.47</td>
<td>0.05</td>
</tr>
<tr>
<td>p-Isopropyltoluene</td>
<td>17.63</td>
<td>0.26</td>
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<td>1,4-Dichlorobenzene</td>
<td>17.63</td>
<td>0.04</td>
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<td>1,2-Dichlorobenzene</td>
<td>17.79</td>
<td>0.05</td>
</tr>
<tr>
<td>n-Butylbenzene</td>
<td>17.95</td>
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</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>18.84</td>
<td>0.20</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>19.07</td>
<td>0.10</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>19.24</td>
<td>0.10</td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>19.24</td>
<td>0.14</td>
</tr>
</tbody>
</table>

<sup>a</sup> Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

<sup>b</sup> MDL based on a 25-mL sample volume.
Esteemed reader,

TABLE 3
ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES

<table>
<thead>
<tr>
<th>Estimated Quantitation Limits</th>
<th>5-mL Ground Water Purge (µg/L)</th>
<th>25-mL Ground water Purge (µg/L)</th>
<th>Low Soil/Sediment&lt;sup&gt;b&lt;/sup&gt; µg/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5</td>
<td>1</td>
<td>5</td>
</tr>
</tbody>
</table>

<sup>a</sup> Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

<sup>b</sup> EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

<table>
<thead>
<tr>
<th>Other Matrices</th>
<th>Factor&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water miscible liquid waste</td>
<td>50</td>
</tr>
<tr>
<td>High concentration soil and sludge</td>
<td>125</td>
</tr>
<tr>
<td>Non-water miscible waste</td>
<td>500</td>
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</tbody>
</table>

<sup>c</sup> EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.
<table>
<thead>
<tr>
<th>m/z</th>
<th>Required Intensity (relative abundance)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>15 to 40% of m/z 95</td>
</tr>
<tr>
<td>75</td>
<td>30 to 60% of m/z 95</td>
</tr>
<tr>
<td>95</td>
<td>Base peak, 100% relative abundance</td>
</tr>
<tr>
<td>96</td>
<td>5 to 9% of m/z 95</td>
</tr>
<tr>
<td>173</td>
<td>Less than 2% of m/z 174</td>
</tr>
<tr>
<td>174</td>
<td>Greater than 50% of m/z 95</td>
</tr>
<tr>
<td>175</td>
<td>5 to 9% of m/z 174</td>
</tr>
<tr>
<td>176</td>
<td>Greater than 95% but less than 101% of m/z 174</td>
</tr>
<tr>
<td>177</td>
<td>5 to 9% of m/z 176</td>
</tr>
</tbody>
</table>

*a Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers’ instructions), provided that method performance is not adversely affected.*
### TABLE 5

**CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Primary Characteristic Ion</th>
<th>Secondary Characteristic Ion(s)</th>
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<tbody>
<tr>
<td>Acetone</td>
<td>58</td>
<td>43</td>
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<tr>
<td>Acetonitrile</td>
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<td>Acrolein</td>
<td>56</td>
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<td>Acrylonitrile</td>
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<tr>
<td>Allyl alcohol</td>
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<td>58, 39</td>
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<tr>
<td>Allyl chloride</td>
<td>76</td>
<td>41, 39, 78</td>
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<tr>
<td>Benzene</td>
<td>78</td>
<td>-</td>
</tr>
<tr>
<td>Benzylic chloride</td>
<td>91</td>
<td>126, 65, 128</td>
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<tr>
<td>Bromoacetone</td>
<td>136</td>
<td>43, 138, 93, 95</td>
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<tr>
<td>Bromobenzene</td>
<td>156</td>
<td>77, 158</td>
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<tr>
<td>Bromochloromethane</td>
<td>128</td>
<td>49, 130</td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>83</td>
<td>85, 127</td>
</tr>
<tr>
<td>Bromoform</td>
<td>173</td>
<td>175, 254</td>
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<tr>
<td>Bromomethane</td>
<td>94</td>
<td>96</td>
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<tr>
<td>iso-Butanol</td>
<td>74</td>
<td>43</td>
</tr>
<tr>
<td>n-Butanol</td>
<td>56</td>
<td>41</td>
</tr>
<tr>
<td>2-Butanone</td>
<td>72</td>
<td>43</td>
</tr>
<tr>
<td>n-Butylbenzene</td>
<td>91</td>
<td>92, 134</td>
</tr>
<tr>
<td>sec-Butylbenzene</td>
<td>105</td>
<td>134</td>
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<td>tert-Butylbenzene</td>
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<td>78</td>
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<tr>
<td>Carbon tetrachloride</td>
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<td>119</td>
</tr>
<tr>
<td>Chloral hydrate</td>
<td>82</td>
<td>44, 84, 86, 111</td>
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<tr>
<td>Chloroacetonitrile</td>
<td>48</td>
<td>75</td>
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<tr>
<td>Chlorobenzene</td>
<td>112</td>
<td>77, 114</td>
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<tr>
<td>1-Chlorobutane</td>
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<td>49</td>
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<tr>
<td>Chlorodibromomethane</td>
<td>129</td>
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<td>64 (49*)</td>
<td>66 (51*)</td>
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<td>49</td>
<td>44, 43, 51, 80</td>
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<td>Bis(2-chloroethyl) sulfide</td>
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<td>111, 158, 160</td>
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<td>65, 106</td>
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<td>Chloroform</td>
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<td>85</td>
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<td>52 (51*)</td>
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<td>3-Chloropropionitrile</td>
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<td>49, 89, 91</td>
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<tr>
<td>2-Chlorotoluene</td>
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<td>126</td>
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<tr>
<td>4-Chlorotoluene</td>
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<td>126</td>
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<tr>
<td>1,2-Dibromo-3-chloropropane</td>
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<td>146</td>
<td>111, 148</td>
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<td>1,4-Dichlorobenzene</td>
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<td>111, 148</td>
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<td>cis-1,4-Dichloro-2-butene</td>
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<td>88, 75</td>
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<td>cis-1,2-Dichloroethene</td>
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<td>trans-1,2-Dichloroethene</td>
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<td>110, 77</td>
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<td>trans-1,3-Dichloropropene</td>
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<td>86, 49</td>
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<td>Methyl iodide</td>
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<td>127, 141</td>
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<td>Secondary Characteristic Ion(s)</td>
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<td>43, 44</td>
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<td>Propionitrile (ethyl cyanide)</td>
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<td>59</td>
<td>41, 39</td>
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<td>n-Propylbenzene</td>
<td>91</td>
<td>120</td>
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<td>Pyridine</td>
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<td>182, 145</td>
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<td>1,3,5-Trimethylbenzene</td>
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<td>120</td>
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<td>91</td>
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<tr>
<td>p-Xylene</td>
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<td>91</td>
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<tr>
<td>Internal Standards/Surrogates:</td>
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<tr>
<td>Benzene-d₆</td>
<td>84</td>
<td>83</td>
</tr>
<tr>
<td>Bromobenzene-d₅</td>
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</tr>
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<td>131</td>
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<td>Chlorobenzene-d₅</td>
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<td>1,1,2-Trichloroethane-d₃</td>
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<tr>
<td>4-Bromofluorobenzene</td>
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<td>174, 176</td>
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<td>Chloroform-d₁</td>
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<td>Compound</td>
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<td>Internal Standards/Surrogates</td>
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<tr>
<td>Toluene-d₈</td>
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<tr>
<td>Pentafluorobenzene</td>
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* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).
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\(^a\) Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

\(^b\) Standard deviation was calculated by pooling data from three concentrations.
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</tr>
<tr>
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<td>7</td>
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</tr>
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<td>1,1,1,2-Tetrachloroethane</td>
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<td>100</td>
<td>4.7</td>
<td>4.7</td>
</tr>
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<td>7</td>
<td>100</td>
<td>12.0</td>
<td>12.0</td>
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<td>96</td>
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<td>5.9</td>
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<td>7</td>
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<td>100</td>
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<td>4.0</td>
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<tr>
<td>1,1,2-Trichloroethane</td>
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<td>7</td>
<td>102</td>
<td>4.9</td>
<td>4.8</td>
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<td>Trichloroethylene</td>
<td>0.1</td>
<td>7</td>
<td>104</td>
<td>2.0</td>
<td>1.9</td>
</tr>
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<td>Trichlorofluoromethane</td>
<td>0.1</td>
<td>7</td>
<td>97</td>
<td>4.6</td>
<td>4.7</td>
</tr>
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<td>1,2,3-Trichloropropane</td>
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<td>7</td>
<td>96</td>
<td>6.5</td>
<td>6.8</td>
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<td>1,2,4-Trimethylbenzene</td>
<td>0.5</td>
<td>7</td>
<td>96</td>
<td>6.5</td>
<td>6.8</td>
</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
<td>0.5</td>
<td>7</td>
<td>101</td>
<td>4.2</td>
<td>4.2</td>
</tr>
<tr>
<td>Vinyl chloride</td>
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<td>104</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>0.5</td>
<td>7</td>
<td>106</td>
<td>7.5</td>
<td>7.1</td>
</tr>
<tr>
<td>m-Xylene</td>
<td>0.5</td>
<td>7</td>
<td>106</td>
<td>4.6</td>
<td>4.3</td>
</tr>
<tr>
<td>p-Xylene</td>
<td>0.5</td>
<td>7</td>
<td>97</td>
<td>6.1</td>
<td>6.3</td>
</tr>
</tbody>
</table>

<sup>a</sup> Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.
### TABLE 8
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

<table>
<thead>
<tr>
<th>Surrogate Compound</th>
<th>Water</th>
<th>Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene&lt;sup&gt;a&lt;/sup&gt;</td>
<td>86-115</td>
<td>74-121</td>
</tr>
<tr>
<td>Dibromofluoromethane&lt;sup&gt;a&lt;/sup&gt;</td>
<td>86-118</td>
<td>80-120</td>
</tr>
<tr>
<td>Toluene-d&lt;sub&gt;8&lt;/sub&gt;&lt;sup&gt;a&lt;/sup&gt;</td>
<td>88-110</td>
<td>81-117</td>
</tr>
<tr>
<td>Dichloroethane-d&lt;sub&gt;4&lt;/sub&gt;&lt;sup&gt;a&lt;/sup&gt;</td>
<td>80-120</td>
<td>80-120</td>
</tr>
</tbody>
</table>

<sup>a</sup> Single laboratory data, for guidance only.

### TABLE 9
QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

<table>
<thead>
<tr>
<th>Approximate Concentration Range (µg/kg)</th>
<th>Volume of Extract&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 - 10,000</td>
<td>100 µL</td>
</tr>
<tr>
<td>1,000 - 20,000</td>
<td>50 µL</td>
</tr>
<tr>
<td>5,000 - 100,000</td>
<td>10 µL</td>
</tr>
<tr>
<td>25,000 - 500,000</td>
<td>100 µL of 1/50 dilution&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Calculate appropriate dilution factor for concentrations exceeding this table.

<sup>a</sup> The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

<sup>b</sup> Dilute an aliquot of the solvent extract and then take 100 µL for analysis.
**TABLE 10**

DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery (%)</th>
<th>%RSD</th>
<th>Blank (ppm)</th>
<th>Spike (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>91</td>
<td>14.8</td>
<td>1.9</td>
<td>5.0</td>
</tr>
<tr>
<td>Benzene</td>
<td>86</td>
<td>21.3</td>
<td>0.1</td>
<td>0.5</td>
</tr>
<tr>
<td>n-Butanol*,**</td>
<td>107</td>
<td>27.8</td>
<td>0.5</td>
<td>5.0</td>
</tr>
<tr>
<td>iso-Butanol*,**</td>
<td>95</td>
<td>19.5</td>
<td>0.9</td>
<td>5.0</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>86</td>
<td>44.7</td>
<td>0.0</td>
<td>0.5</td>
</tr>
<tr>
<td>Carbon disulfide**</td>
<td>53</td>
<td>22.3</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>81</td>
<td>29.3</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Chloroform</td>
<td>84</td>
<td>29.3</td>
<td>0.0</td>
<td>6.0</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>98</td>
<td>24.9</td>
<td>0.0</td>
<td>7.5</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>101</td>
<td>23.1</td>
<td>0.0</td>
<td>0.5</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>97</td>
<td>45.3</td>
<td>0.0</td>
<td>0.7</td>
</tr>
<tr>
<td>Diethyl ether</td>
<td>76</td>
<td>24.3</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>113</td>
<td>27.4</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>83</td>
<td>30.1</td>
<td>0.2</td>
<td>5.0</td>
</tr>
<tr>
<td>Hexachloroethane</td>
<td>71</td>
<td>30.3</td>
<td>0.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>98</td>
<td>45.3</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Methyl ethyl ketone</td>
<td>79</td>
<td>24.6</td>
<td>0.4</td>
<td>5.0</td>
</tr>
<tr>
<td>MIBK</td>
<td>93</td>
<td>31.4</td>
<td>0.0</td>
<td>5.0</td>
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<td>Nitrobenzene</td>
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<td>2.0</td>
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<td>Pyridine</td>
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<td>35.9</td>
<td>0.0</td>
<td>5.0</td>
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<td>Tetrachloroethene</td>
<td>82</td>
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<td>Trichlorofluoromethane</td>
<td>76</td>
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<td>1,1,2-Trichlorotrifluoroethane</td>
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<tr>
<td>Toluene</td>
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<tr>
<td>Trichloroethene</td>
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<td>Vinyl chloride</td>
<td>63</td>
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<td>0.2</td>
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<tr>
<td>α-Xylene</td>
<td>83</td>
<td>29.5</td>
<td>0.4</td>
<td>5.0</td>
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<tr>
<td>m/p-Xylene</td>
<td>84</td>
<td>29.5</td>
<td>0.6</td>
<td>10.0</td>
</tr>
</tbody>
</table>

* Alternate mass employed
** IS quantitation

Data are taken from Reference 9.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery (%)</th>
<th>%RSD</th>
<th>Blank (ppm)</th>
<th>Spike (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone**</td>
<td>105</td>
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<td>2.0</td>
<td>5.0</td>
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<td>44</td>
<td>14</td>
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<td>Benzene-d₆</td>
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<td>44</td>
<td>2.9</td>
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<tr>
<td>n-Butanol**</td>
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<td>71</td>
<td>12</td>
<td>5.0</td>
</tr>
<tr>
<td>iso-Butanol*,**,</td>
<td>132</td>
<td>27</td>
<td>0</td>
<td>5.0</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
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<td>0.5</td>
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<tr>
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<td>0.5</td>
</tr>
<tr>
<td>Carbon disulfide**</td>
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<td>63</td>
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<td>5.0</td>
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<td>71</td>
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<td>5.0</td>
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<td>3.4</td>
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<td>45</td>
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<td>3.0</td>
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</tr>
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<td>Methyl ethyl ketone**</td>
<td>107</td>
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<td>0</td>
<td>0.7</td>
</tr>
<tr>
<td>Trichlorofluoromethane**</td>
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<td>70</td>
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<td>5.0</td>
</tr>
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<td>44</td>
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<td>5.0</td>
</tr>
<tr>
<td>Trichloroethene</td>
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<td>0.5</td>
</tr>
<tr>
<td>Trichloroethene-d₁</td>
<td>55</td>
<td>44</td>
<td>2.8</td>
<td>0.5</td>
</tr>
<tr>
<td>Compound</td>
<td>Recovery (%)</td>
<td>%RSD</td>
<td>Blank (ppm)</td>
<td>Spike (ppm)</td>
</tr>
<tr>
<td>-------------------</td>
<td>--------------</td>
<td>------</td>
<td>-------------</td>
<td>-------------</td>
</tr>
<tr>
<td>Vinyl chloride**</td>
<td>100</td>
<td>69</td>
<td>0</td>
<td>0.2</td>
</tr>
<tr>
<td>o-Xylene</td>
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<td>44</td>
<td>105</td>
<td>5.0</td>
</tr>
<tr>
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<td>44</td>
<td>4.2</td>
<td>5.0</td>
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<tr>
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<td>2583</td>
<td>44</td>
<td>253</td>
<td>10.0</td>
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<td>67</td>
<td>44</td>
<td>3.7</td>
<td>10.0</td>
</tr>
</tbody>
</table>

* Alternate mass employed  
** IS quantitation  
ND = Not Detected  

Data are based on seven measurements and are taken from Reference 9.
### TABLE 12

**METHOD DETECTION LIMITS (METHOD 5031)**

<table>
<thead>
<tr>
<th>Compound</th>
<th>MDL (µg/L)</th>
<th>Concentration Factor</th>
</tr>
</thead>
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<td>Macro a</td>
<td>Macro Micro</td>
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<td>Acetone</td>
<td>31</td>
<td>25-500 -</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>57</td>
<td>25-500 200</td>
</tr>
<tr>
<td>Acrolein</td>
<td>-</td>
<td>- 100</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>16</td>
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</tr>
<tr>
<td>Allyl Alcohol</td>
<td>7</td>
<td>25-500 -</td>
</tr>
<tr>
<td>1-Butanol</td>
<td>-</td>
<td>- 250</td>
</tr>
<tr>
<td>Crotonaldehyde</td>
<td>12</td>
<td>25-500 -</td>
</tr>
<tr>
<td>1,4-Dioxane</td>
<td>12</td>
<td>25-500 150</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>-</td>
<td>- 100</td>
</tr>
<tr>
<td>Isobutyl alcohol</td>
<td>7</td>
<td>25-500 -</td>
</tr>
<tr>
<td>Methanol</td>
<td>38</td>
<td>25-500 140</td>
</tr>
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<td>25-500 -</td>
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<tr>
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<td>Paraldehyde</td>
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<td>25-500 -</td>
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<td>4</td>
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<tr>
<td>o-Toluidine</td>
<td>13</td>
<td>25-500 -</td>
</tr>
</tbody>
</table>

---

a  Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

\[
MDL = 3.134 \times \text{Std. Dev. of low concentration spike (ppb)}.
\]

b  When a 40-mL sample is used, and the first 100 µL of distillate are collected.
# TABLE 13

## TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

<table>
<thead>
<tr>
<th>Target Compound</th>
<th>Surrogate</th>
<th>Internal Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>d$_6$-Acetone</td>
<td>d$_8$-Isopropyl alcohol</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>d$_3$-Acetonitrile</td>
<td>d$_6$-Isopropyl alcohol</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>d$_8$-Isopropyl alcohol</td>
<td></td>
</tr>
<tr>
<td>Allyl alcohol</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
<tr>
<td>Crotonaldehyde</td>
<td>d$_8$-Isopropyl alcohol</td>
<td></td>
</tr>
<tr>
<td>1,4-Dioxane</td>
<td>d$_8$-1,4-Dioxane</td>
<td>d$_7$-Dimethyl formamide</td>
</tr>
<tr>
<td>Isobutyl alcohol</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
<tr>
<td>Methanol</td>
<td>d$_3$-Methanol</td>
<td>d$_4$-Isopropyl alcohol</td>
</tr>
<tr>
<td>Methyl ethyl ketone</td>
<td>d$_8$-Isopropyl alcohol</td>
<td></td>
</tr>
<tr>
<td>N-Nitroso-di-n-butylamine</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
<tr>
<td>Paraldehyde</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
<tr>
<td>2-Picoline</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
<tr>
<td>Propionitrile</td>
<td>d$_8$-Isopropyl alcohol</td>
<td></td>
</tr>
<tr>
<td>Pyridine</td>
<td>d$_8$-Pyridine</td>
<td>d$_7$-Dimethyl formamide</td>
</tr>
<tr>
<td>o-Toluidine</td>
<td>d$_7$-Dimethyl formamide</td>
<td></td>
</tr>
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### TABLE 14
RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Concentration(s) (ng/µL)</th>
</tr>
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</tr>
<tr>
<td>d₅-benzyl alcohol</td>
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</tr>
<tr>
<td>d₁₄-Diglyme</td>
<td>10.0</td>
</tr>
<tr>
<td>d₇-Dimethyl formamide</td>
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</tr>
<tr>
<td>d₈-Isopropyl alcohol</td>
<td>10.0</td>
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<tr>
<td><strong>Surrogates</strong></td>
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</tr>
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<td>d₆-Acetone</td>
<td>10.0</td>
</tr>
<tr>
<td>d₆-Acetonitrile</td>
<td>10.0</td>
</tr>
<tr>
<td>d₆-1,4-Dioxane</td>
<td>10.0</td>
</tr>
<tr>
<td>d₇-Methanol</td>
<td>10.0</td>
</tr>
<tr>
<td>d₅-Pyridine</td>
<td>10.0</td>
</tr>
<tr>
<td><strong>Target Compounds</strong></td>
<td></td>
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<tr>
<td>Acetone</td>
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<tr>
<td>Acetonitrile</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
</tr>
<tr>
<td>Allyl alcohol</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
</tr>
<tr>
<td>Crotonaldehyde</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
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<tr>
<td>Isobutyl alcohol</td>
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<td>Methanol</td>
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<tr>
<td>Methyl ethyl ketone</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
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</tr>
<tr>
<td>Paraldehyde</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
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<tr>
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<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
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<tr>
<td>Propionitrile</td>
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<tr>
<td>Pyridine</td>
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<td>α-Toluidine</td>
<td>1.0, 5.0, 10.0, 25.0, 100.0</td>
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<td>Compound</td>
<td>Quantitation Ion</td>
</tr>
<tr>
<td>---------------------------</td>
<td>------------------</td>
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<td>Internal Standards</td>
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<td>d₁₄-Diglyme</td>
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<td>d₂-Dimethyl formamide</td>
<td>50</td>
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<tr>
<td>Surrogates</td>
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</tr>
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<td>d₅-Acetone</td>
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<tr>
<td>d₃-Methanol</td>
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<tr>
<td>d₅-Acetonitrile</td>
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<tr>
<td>d₅-1,4-Dioxane</td>
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<tr>
<td>d₅-Pyridine</td>
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<td>d₅-Phenol</td>
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<td>Target Compounds</td>
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<td>Methanol</td>
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<td>Methyl ethyl ketone</td>
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<tr>
<td>Methacrylonitrile</td>
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<tr>
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<tr>
<td>Methyl isobutyl ketone</td>
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<tr>
<td>Propionitrile</td>
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<tr>
<td>Crotonaldehyde</td>
<td>41</td>
</tr>
<tr>
<td>1,4-Dioxane</td>
<td>58</td>
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<tr>
<td>Paraldehyde</td>
<td>45</td>
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<tr>
<td>Isobutyl alcohol</td>
<td>43</td>
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<tr>
<td>Allyl alcohol</td>
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<tr>
<td>Pyridine</td>
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<tr>
<td>2-Picoline</td>
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<tr>
<td>N-Nitroso-di-n-butylamine</td>
<td>84</td>
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<tr>
<td>Aniline</td>
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<tr>
<td>α-Toluidine</td>
<td>106</td>
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<tr>
<td>Phenol</td>
<td>94</td>
</tr>
</tbody>
</table>

a These ions were used for quantitation in selected ion monitoring.
b GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.
c Compound removed from target analyte list due to poor accuracy and precision.
TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION\textsuperscript{a} (METHOD 5031 - MACRODISTILLATION TECHNIQUE) 
(Single Laboratory and Single Operator)

<table>
<thead>
<tr>
<th>Compound</th>
<th>25 ppb Spike</th>
<th>100 ppb Spike</th>
<th>500 ppb Spike</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean %R</td>
<td>%RSD</td>
<td>Mean %R</td>
</tr>
<tr>
<td>d$_6$-Acetone</td>
<td>66 24</td>
<td>69 14</td>
<td>65 16</td>
</tr>
<tr>
<td>d$_3$-Acetonitrile</td>
<td>89 18</td>
<td>80 18</td>
<td>70 10</td>
</tr>
<tr>
<td>d$_8$-1,4-Dioxane</td>
<td>56 34</td>
<td>58 11</td>
<td>61 18</td>
</tr>
<tr>
<td>d$_3$-Methanol</td>
<td>43 29</td>
<td>48 19</td>
<td>56 14</td>
</tr>
<tr>
<td>d$_5$-Pyridine</td>
<td>83 6.3</td>
<td>84 7.8</td>
<td>85 9.0</td>
</tr>
<tr>
<td>Acetone</td>
<td>67 45</td>
<td>63 14</td>
<td>60 14</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>44 35</td>
<td>52 15</td>
<td>56 15</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>49 42</td>
<td>47 27</td>
<td>45 27</td>
</tr>
<tr>
<td>Allyl alcohol</td>
<td>69 13</td>
<td>70 9.7</td>
<td>73 10</td>
</tr>
<tr>
<td>Crotonaldehyde</td>
<td>68 22</td>
<td>68 13</td>
<td>69 13</td>
</tr>
<tr>
<td>1,4-Dioxane</td>
<td>63 25</td>
<td>55 16</td>
<td>54 13</td>
</tr>
<tr>
<td>Isobutyl alcohol</td>
<td>66 14</td>
<td>66 5.7</td>
<td>65 7.9</td>
</tr>
<tr>
<td>Methanol</td>
<td>50 36</td>
<td>46 22</td>
<td>49 18</td>
</tr>
<tr>
<td>Methyl ethyl ketone</td>
<td>55 37</td>
<td>56 20</td>
<td>52 19</td>
</tr>
<tr>
<td>N-Nitroso-di-n-butylamine</td>
<td>57 21</td>
<td>61 15</td>
<td>72 18</td>
</tr>
<tr>
<td>Paraldehyde</td>
<td>65 20</td>
<td>66 11</td>
<td>60 8.9</td>
</tr>
<tr>
<td>Picoline</td>
<td>81 12</td>
<td>81 6.8</td>
<td>84 8.0</td>
</tr>
<tr>
<td>Propionitrile</td>
<td>67 22</td>
<td>69 13</td>
<td>68 13</td>
</tr>
<tr>
<td>Pyridine</td>
<td>74 7.4</td>
<td>72 6.7</td>
<td>74 7.3</td>
</tr>
<tr>
<td>o-Toluidine</td>
<td>52 31</td>
<td>54 15</td>
<td>58 12</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.
### TABLE 17

**RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery per Replicate (ng)</th>
<th></th>
<th></th>
<th></th>
<th>Mean</th>
<th>RSD</th>
<th>Rec</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>8.0</td>
<td>7.5</td>
<td>6.7</td>
<td>5.4</td>
<td>6.6</td>
<td>6.8</td>
<td>13.0</td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>13.3</td>
<td>16.5</td>
<td>14.9</td>
<td>13.0</td>
<td>10.3</td>
<td>13.6</td>
<td>15.2</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>17.1</td>
<td>16.7</td>
<td>15.1</td>
<td>14.8</td>
<td>15.6</td>
<td>15.9</td>
<td>5.7</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>24.5</td>
<td>22.7</td>
<td>19.7</td>
<td>19.4</td>
<td>20.6</td>
<td>21.4</td>
<td>9.1</td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td>22.7</td>
<td>23.6</td>
<td>19.4</td>
<td>18.3</td>
<td>20.1</td>
<td>20.8</td>
<td>0.7</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>18.3</td>
<td>18.0</td>
<td>16.7</td>
<td>15.6</td>
<td>15.9</td>
<td>16.9</td>
<td>6.4</td>
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<tr>
<td>cis-1,2-Dichloroethene</td>
<td>26.1</td>
<td>23.1</td>
<td>22.6</td>
<td>20.3</td>
<td>20.8</td>
<td>22.6</td>
<td>9.0</td>
</tr>
<tr>
<td>Bromochloromethane</td>
<td>24.5</td>
<td>25.4</td>
<td>20.9</td>
<td>20.1</td>
<td>20.1</td>
<td>22.2</td>
<td>10.2</td>
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<tr>
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<td>26.5</td>
<td>26.0</td>
<td>22.1</td>
<td>18.9</td>
<td>22.1</td>
<td>23.1</td>
<td>12.2</td>
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<td>23.0</td>
<td>23.9</td>
<td>16.7</td>
<td>31.2</td>
<td>23.4</td>
<td>12.2</td>
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<td>24.2</td>
<td>22.6</td>
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<td>23.3</td>
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<td>20.5</td>
<td>19.2</td>
<td>14.4</td>
<td>19.1</td>
<td>18.9</td>
<td>12.7</td>
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<td>23.1</td>
<td>19.0</td>
<td>23.3</td>
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<td>23.6</td>
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<td>23.6</td>
<td>23.6</td>
<td>16.9</td>
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<td>24.5</td>
<td>21.6</td>
<td>17.7</td>
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<td>12.1</td>
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<td>22.7</td>
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<td>22.2</td>
<td>22.3</td>
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<td>22.0</td>
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<td>20.9</td>
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<td>15.7</td>
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<td>25.6</td>
<td>26.0</td>
<td>20.1</td>
<td>23.5</td>
<td>24.4</td>
<td>9.9</td>
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<td>25.1</td>
<td>24.2</td>
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<td>22.9</td>
<td>16.6</td>
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<td>21.8</td>
<td>20.0</td>
<td>15.5</td>
<td>19.1</td>
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<td>10.7</td>
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<td>23.0</td>
<td>25.6</td>
<td>15.9</td>
<td>21.4</td>
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<td>15.8</td>
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<td>13.9</td>
<td>21.9</td>
<td>21.6</td>
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<td>13.0</td>
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<td>4-Chlorotoluene</td>
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<td>12.1</td>
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### TABLE 19

**RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)**

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<td>7.9</td>
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Data in Table 19 are from Reference 15.
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<th>Compound</th>
<th>Soil/H₂O&lt;sup&gt;b&lt;/sup&gt; Recovery</th>
<th>Soil/Oil&lt;sup&gt;c&lt;/sup&gt; Recovery</th>
<th>Soil/Oil/H₂O Recovery</th>
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<td>Mean</td>
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<td>Bromomethane</td>
<td>58</td>
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<td>47</td>
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<td>46</td>
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<tr>
<td>Chloroethane</td>
<td>46</td>
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<td>41</td>
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<td>65</td>
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<td>Acetone</td>
<td>INT&lt;sup&gt;e&lt;/sup&gt;</td>
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<td>9</td>
<td>47</td>
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<td>6</td>
<td>58</td>
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<td>trans-1,2-Trichloroethane</td>
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<td>60</td>
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<td>93</td>
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<tr>
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<td>50</td>
<td>117</td>
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<tr>
<td>2-Butanone</td>
<td>INT</td>
<td>36</td>
<td>38</td>
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<td>1,1,1-Trichloroethane</td>
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<td>13</td>
<td>72</td>
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<tr>
<td>Carbon tetrachloride</td>
<td>172</td>
<td>122</td>
<td>INT</td>
</tr>
<tr>
<td>Vinyl acetate</td>
<td>88</td>
<td>11</td>
<td>INT</td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>93</td>
<td>4</td>
<td>91</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
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<td>50</td>
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<td>1,2-Dichloropropane</td>
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<td>102</td>
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<tr>
<td>trans-1,3-Dichloropropene</td>
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<td>84</td>
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<td>Trichloroethene</td>
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<td>Dibromochloromethane</td>
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<td>1,1,2-Trichloroethane</td>
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<td>72</td>
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<td>Benzene</td>
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<tr>
<td>cis-1,3-Dichloropropene</td>
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<td>12</td>
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<td>Chlorobenzene</td>
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<td>93</td>
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<tr>
<td>Ethylbenzene</td>
<td>114</td>
<td>13</td>
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<tr>
<td>Styrene</td>
<td>106</td>
<td>8</td>
<td>93</td>
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<tr>
<td>p-Xylene</td>
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<td>9</td>
<td>CONT</td>
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<tr>
<td>o-Xylene</td>
<td>105</td>
<td>8</td>
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TABLE 20 (cont.)

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<th>Compound</th>
<th>Soil/H₂O&lt;sup&gt;a&lt;/sup&gt; Recovery Mean</th>
<th>Soil/Oil&lt;sup&gt;c&lt;/sup&gt; Recovery Mean</th>
<th>Soil/Oil/H₂O Recovery Mean</th>
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<tr>
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<td>RSD</td>
<td>RSD</td>
<td>RSD</td>
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<td>1,2-Dichloroethane</td>
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<td>151</td>
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<td>Toluene-d₈</td>
<td>96</td>
<td>79</td>
<td>82</td>
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<tr>
<td>Bromofluorobenzene</td>
<td>139</td>
<td>37</td>
<td>62</td>
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</table>

Surrogates

- a Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

- b Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

- c Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

- d Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

- e Interference by co-eluting compounds prevented accurate measurement of analyte.

- f Contamination of sample matrix by analyte prevented assessment of efficiency.
TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)*

<table>
<thead>
<tr>
<th>Compound</th>
<th>Efficiency</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean (%)</td>
<td>RSD (%)</td>
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</tr>
<tr>
<td>Chloromethane</td>
<td>N/A^b</td>
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</tr>
<tr>
<td>Bromomethane</td>
<td>N/A^b</td>
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</tr>
<tr>
<td>Vinyl chloride</td>
<td>N/A^b</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chloroethane</td>
<td>N/A^b</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>CONT^c</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>CONT^c</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbon disulfide</td>
<td>79</td>
<td>36</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>122</td>
<td>39</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>126</td>
<td>35</td>
<td></td>
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<tr>
<td>trans-1,2-Trichloroethene</td>
<td>109</td>
<td>46</td>
<td></td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene</td>
<td>106</td>
<td>22</td>
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</tr>
<tr>
<td>Chloroform</td>
<td>111</td>
<td>32</td>
<td></td>
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<td>117</td>
<td>27</td>
<td></td>
</tr>
<tr>
<td>2-Butanone</td>
<td>INT^d</td>
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<td></td>
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<td>1,1,1-Trichloroethane</td>
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<td>30</td>
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<tr>
<td>Carbon tetrachloride</td>
<td>83</td>
<td>34</td>
<td></td>
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<tr>
<td>Vinyl acetate</td>
<td>INT^d</td>
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<td></td>
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<tr>
<td>Bromodichloromethane</td>
<td>97</td>
<td>22</td>
<td></td>
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<td>67</td>
<td>20</td>
<td></td>
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<tr>
<td>1,2-Dichloropropane</td>
<td>117</td>
<td>23</td>
<td></td>
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<td>trans-1,3-Dichloropropene</td>
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<td>22</td>
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<td>Trichloroethene</td>
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<td>1,1,2-Trichloroethane</td>
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<td>Benzene</td>
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<td>cis-1,3-Dichloropropene</td>
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<td>Bromoform</td>
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<td>4-Methyl-2-pentanone</td>
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<td>Styrene</td>
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<td>p-Xylene</td>
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<td>o-Xylene</td>
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### TABLE 21 (cont.)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Efficiency</th>
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<tbody>
<tr>
<td></td>
<td>Mean (%)</td>
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<td>Surrogates</td>
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<td>Toluene-d$_8$</td>
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<tr>
<td>Bromofluorobenzene</td>
<td>52</td>
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</table>

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- Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

- No analyses.

- Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

- Interfering by co-eluting compounds prevented accurate measurement of analyte.
## TABLE 22

**METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Method Detection Limit (ppb)</th>
<th>External Standard Method</th>
<th>Internal Standard Method</th>
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<tbody>
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<td>9.2</td>
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</tr>
<tr>
<td>Methylene chloride</td>
<td>CONT&lt;sup&gt;b&lt;/sup&gt;</td>
<td>CONT&lt;sup&gt;b&lt;/sup&gt;</td>
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</tr>
<tr>
<td>Acetone</td>
<td>CONT&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>Carbon disulfide</td>
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<tr>
<td>2-Butanone</td>
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<td>INT&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>Carbon tetrachloride</td>
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</tr>
<tr>
<td>Vinyl acetate</td>
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<td>INT&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>Benzene</td>
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<td>cis-1,3-Dichloropropene</td>
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<tr>
<td>Bromoform</td>
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<td>4.0</td>
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<td>7.7</td>
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<td>4-Methyl-2-pentanone</td>
<td>7.5</td>
<td>8.0</td>
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<tr>
<td>Tetrachloroethene</td>
<td>4.3</td>
<td>4.0</td>
<td></td>
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<td>2.5</td>
<td></td>
</tr>
<tr>
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<tr>
<td>Ethylbenzene</td>
<td>3.6</td>
<td>3.5</td>
<td></td>
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<tr>
<td>Styrene</td>
<td>3.5</td>
<td>3.3</td>
<td></td>
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<tr>
<td>p-Xylene</td>
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<tr>
<td>o-Xylene</td>
<td>3.3</td>
<td>4.7</td>
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</table>

Footnotes are on the following page.
Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked at 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

Contamination of sample by analyte prevented determination.

Interference by co-eluting compounds prevented accurate quantitation.
TABLE 23
VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER USING VACUUM DISTILLATION (METHOD 5032)\textsuperscript{a}

<table>
<thead>
<tr>
<th>Compound</th>
<th>5 mL H\textsubscript{2}O\textsuperscript{b} Recovery</th>
<th>20 mL H\textsubscript{2}O\textsuperscript{c} Recovery</th>
<th>20 mL H\textsubscript{2}O/Oil Recovery</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>RSD</td>
<td>Mean</td>
</tr>
<tr>
<td>Chloromethane</td>
<td>114</td>
<td>27</td>
<td>116</td>
</tr>
<tr>
<td>Bromomethane</td>
<td>131</td>
<td>14</td>
<td>121</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>131</td>
<td>13</td>
<td>120</td>
</tr>
<tr>
<td>Chloroethane</td>
<td>110</td>
<td>15</td>
<td>99</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>87</td>
<td>16</td>
<td>105</td>
</tr>
<tr>
<td>Acetone</td>
<td>83</td>
<td>22</td>
<td>65</td>
</tr>
<tr>
<td>Carbon disulfide</td>
<td>138</td>
<td>17</td>
<td>133</td>
</tr>
<tr>
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<td>105</td>
<td>11</td>
<td>89</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>118</td>
<td>10</td>
<td>119</td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td>105</td>
<td>11</td>
<td>107</td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene</td>
<td>106</td>
<td>7</td>
<td>99</td>
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<td>Chloroform</td>
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<td>104</td>
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<td>1,2-Dichloroethane</td>
<td>104</td>
<td>6</td>
<td>109</td>
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<tr>
<td>2-Butanone</td>
<td>83</td>
<td>50</td>
<td>106</td>
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<td>1,1,1-Trichloroethane</td>
<td>118</td>
<td>9</td>
<td>109</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>102</td>
<td>6</td>
<td>108</td>
</tr>
<tr>
<td>Vinyl acetate</td>
<td>90</td>
<td>16</td>
<td>99</td>
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<tr>
<td>Bromodichloromethane</td>
<td>104</td>
<td>3</td>
<td>110</td>
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<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>85</td>
<td>17</td>
<td>81</td>
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<td>100</td>
<td>6</td>
<td>103</td>
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<td>105</td>
<td>8</td>
<td>105</td>
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<td>Trichloroethene</td>
<td>98</td>
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<td>7</td>
<td>100</td>
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<tr>
<td>Benzene</td>
<td>97</td>
<td>4</td>
<td>100</td>
</tr>
<tr>
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<td>106</td>
<td>5</td>
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<tr>
<td>Bromoform</td>
<td>93</td>
<td>16</td>
<td>94</td>
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<td>2-Hexanone</td>
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<td>63</td>
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<td>4-Methyl-2-pentanone</td>
<td>79</td>
<td>24</td>
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<td>Tetrachloroethene</td>
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<td>3</td>
<td>97</td>
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<tr>
<td>Toluene</td>
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<td>6</td>
<td>97</td>
</tr>
<tr>
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<td>6</td>
<td>98</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>100</td>
<td>3</td>
<td>92</td>
</tr>
<tr>
<td>Styrene</td>
<td>98</td>
<td>4</td>
<td>97</td>
</tr>
<tr>
<td>p-Xylene</td>
<td>96</td>
<td>4</td>
<td>94</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>96</td>
<td>7</td>
<td>95</td>
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</table>
### TABLE 23 (cont.)

<table>
<thead>
<tr>
<th>Compound</th>
<th>5 mL H₂O&lt;sup&gt;a&lt;/sup&gt; Recovery Mean</th>
<th>5 mL H₂O&lt;sup&gt;a&lt;/sup&gt; Recovery RSD</th>
<th>20 mL H₂O&lt;sup&gt;c&lt;/sup&gt; Recovery Mean</th>
<th>20 mL H₂O&lt;sup&gt;c&lt;/sup&gt; Recovery RSD</th>
<th>20 mL H₂O/Oil Recovery Mean</th>
<th>20 mL H₂O/Oil Recovery RSD</th>
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<td>7</td>
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<td>106</td>
<td>9</td>
<td>40</td>
<td>8</td>
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<sup>a</sup> Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

<sup>b</sup> Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

<sup>c</sup> Interference by co-eluting compounds prevented accurate assessment of recovery.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Water (µg/L)</th>
<th>Soil (µg/kg)</th>
<th>Tissue (µg/kg)</th>
<th>Oil (mg/kg)</th>
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<tr>
<td>Chloromethane</td>
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<td>7.3</td>
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</tr>
<tr>
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<td>4.9</td>
<td>9.8</td>
<td>N/A</td>
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<tr>
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<td>6.0</td>
<td>9.4</td>
<td>N/A</td>
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<td>6.0</td>
<td>10.0</td>
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<tr>
<td>Methylene chloride</td>
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<td>4.0</td>
<td>CONT</td>
<td>0.05</td>
</tr>
<tr>
<td>Acetone</td>
<td>5.6</td>
<td>CONT</td>
<td>CONT</td>
<td>0.06</td>
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<tr>
<td>Carbon disulfide</td>
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<td>2.0</td>
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<td>INT</td>
<td>INT</td>
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<td>1,1,1-Trichloroethane</td>
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<td>1.7</td>
<td>4.2</td>
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<td>Carbon tetrachloride</td>
<td>1.4</td>
<td>1.5</td>
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<td>0.13</td>
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<td>Vinyl acetate</td>
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<td>INT</td>
<td>INT</td>
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<tr>
<td>Bromodichloromethane</td>
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<td>1.4</td>
<td>2.8</td>
<td>0.06</td>
</tr>
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<td>3.7</td>
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<td>1.7</td>
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<td>0.05</td>
</tr>
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<td>1.7</td>
<td>4.0</td>
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<td>Dibromochloromethane</td>
<td>1.7</td>
<td>1.5</td>
<td>3.2</td>
<td>0.07</td>
</tr>
<tr>
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<td>1.7</td>
<td>3.3</td>
<td>0.05</td>
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<td>1.5</td>
<td>3.2</td>
<td>0.05</td>
</tr>
<tr>
<td>cis-1,3-Dichloropropene</td>
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<td>1.7</td>
<td>3.0</td>
<td>0.04</td>
</tr>
<tr>
<td>Bromoform</td>
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<td>1.5</td>
<td>4.0</td>
<td>0.05</td>
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<td>3.6</td>
<td>8.0</td>
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<tr>
<td>4-Methyl-2-pentanone</td>
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<td>4.6</td>
<td>8.0</td>
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<tr>
<td>Tetrachloroethene</td>
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<td>1.6</td>
<td>4.0</td>
<td>0.10</td>
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<td>1.4</td>
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<td>0.06</td>
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<tr>
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<td>2.8</td>
<td>3.5</td>
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<tr>
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<td>1.4</td>
<td>3.3</td>
<td>0.18</td>
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<td>p-Xylene</td>
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<td>2.9</td>
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<tr>
<td>o-Xylene</td>
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<td>3.4</td>
<td>4.7</td>
<td>0.07</td>
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</table>

Footnotes are found on the following page.
<table>
<thead>
<tr>
<th></th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.</td>
</tr>
<tr>
<td>b</td>
<td>Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.</td>
</tr>
<tr>
<td>c</td>
<td>Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.</td>
</tr>
<tr>
<td>d</td>
<td>Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.</td>
</tr>
<tr>
<td>e</td>
<td>No analyses.</td>
</tr>
<tr>
<td>f</td>
<td>Contamination of sample by analyte prevented determination.</td>
</tr>
<tr>
<td>g</td>
<td>Interference by co-eluting compounds prevented accurate quantitation.</td>
</tr>
</tbody>
</table>
### TABLE 25

**METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES**  
(METHOD 5032) (EXTERNAL STANDARD METHOD)*

<table>
<thead>
<tr>
<th>Compound</th>
<th>Water&lt;sup&gt;b&lt;/sup&gt; (µg/L)</th>
<th>Soil&lt;sup&gt;c&lt;/sup&gt; (µg/kg)</th>
<th>Tissue&lt;sup&gt;d&lt;/sup&gt; (µg/kg)</th>
<th>Oil&lt;sup&gt;e&lt;/sup&gt; (mg/kg)</th>
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</thead>
<tbody>
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<td>Chloromethane</td>
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<td>7.8</td>
<td>N/A&lt;sup&gt;g&lt;/sup&gt;</td>
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<tr>
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<td>4.9&lt;sup&gt;f&lt;/sup&gt;</td>
<td>9.7</td>
<td>N/A&lt;sup&gt;g&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>4.0</td>
<td>7.1&lt;sup&gt;i&lt;/sup&gt;</td>
<td>9.5</td>
<td>N/A&lt;sup&gt;g&lt;/sup&gt;</td>
</tr>
<tr>
<td>Chloroethane</td>
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<td>9.2</td>
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<td>Methylene chloride</td>
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<td>3.3&lt;sup&gt;j&lt;/sup&gt;</td>
<td>CONT&lt;sup&gt;h&lt;/sup&gt;</td>
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<td>33.0&lt;sup&gt;j&lt;/sup&gt;</td>
<td>CONT&lt;sup&gt;h&lt;/sup&gt;</td>
<td>CONT&lt;sup&gt;h&lt;/sup&gt;</td>
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<td>3.8</td>
<td>4.0</td>
<td>0.19</td>
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<tr>
<td>1,1-Dichloroethane</td>
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<td>4.0</td>
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<td>1.7</td>
<td>3.3</td>
<td>0.06</td>
</tr>
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<td>2-Butanone</td>
<td>57.0&lt;sup&gt;j&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
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<td>2.4</td>
<td>1.1</td>
<td>0.08</td>
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<tr>
<td>Carbon tetrachloride</td>
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<td>1.7</td>
<td>3.2</td>
<td>0.15</td>
</tr>
<tr>
<td>Vinyl acetate</td>
<td>23.0&lt;sup&gt;j&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
<td>INT&lt;sup&gt;i&lt;/sup&gt;</td>
</tr>
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<td>1,2-Dichloropropane</td>
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<td>3.7</td>
<td>3.8</td>
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<tr>
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<td>2.9</td>
<td>3.6</td>
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<td>4.4</td>
<td>3.0</td>
<td>0.09</td>
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<tr>
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<td>2.6</td>
<td>3.3</td>
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<td>3.3</td>
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</table>
Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.

Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.

These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.

These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.

Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.

Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.

No analyses.

Contamination of sample by analyte prevented determination.

Interference by co-eluting compound prevented accurate quantitation.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean (%)</td>
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<tr>
<td>Chloromethane</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Bromomethane</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Chloroethane</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>Methylene chloride</td>
<td>62</td>
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<tr>
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<td>Carbon disulfide</td>
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<tr>
<td>1,1-Dichloroethene</td>
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<tr>
<td>1,1-Dichloroethane</td>
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<tr>
<td>trans-1,2-Trichloroethene</td>
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<tr>
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<tr>
<td>2-Butanone</td>
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<td>Carbon tetrachloride</td>
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<td>Vinyl acetate</td>
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TABLE 26 (cont.)

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<th>Compound</th>
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Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

- Not analyzed.
- Interference by co-evaluating compounds prevented accurate measurement of analyte.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Method Detection Limit (ppb)</th>
<th>Method Detection Limit (ppb)</th>
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</thead>
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<td>Internal Standard Method</td>
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<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>Bromomethane</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>Vinyl chloride</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
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<td>Chloroethane</td>
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<td>N/A&lt;sup&gt;b&lt;/sup&gt;</td>
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<td>Chloroform</td>
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<td>2-Butanone</td>
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<td>INT&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>1,2-Dichloropropane</td>
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<td>INT&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>4-Methyl-2-pentanone</td>
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<td>INT&lt;sup&gt;c&lt;/sup&gt;</td>
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TABLE 27 (cont.)

<p>| |</p>
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<td><strong>Method detection limits</strong> are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.</td>
</tr>
<tr>
<td><strong>b</strong></td>
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<tr>
<td>No analyses.</td>
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<tr>
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<td>Interference by co-eluting compounds prevented accurate quantitation.</td>
</tr>
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<td><strong>Table 28</strong></td>
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<td>INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS</td>
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<td>Vinyl chloride</td>
<td>Carbon tetrachloride</td>
<td>Styrene</td>
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<td>Benzene</td>
<td>iso-Propylbenzene</td>
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<td>Dibromomethane</td>
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<td>Trichlorofluoromethane</td>
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<td>n-Propylbenzene</td>
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<td>tert-Butylbenzene</td>
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<td>1,2,4-Trimethylbenzene</td>
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<tr>
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<td>Toluene</td>
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<td>1,3-Dichlorobenzene</td>
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<td>o-Xylene</td>
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CD-ROM 8260B-73 Revision 2 December 1996
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<th>% RSD</th>
<th>MDL (µg/kg)</th>
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<td>0.21</td>
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<td>Hexachlorobutadiene</td>
<td>4.1</td>
<td>0.38</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>8.2</td>
<td>0.62</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>16.8</td>
<td>3.4</td>
</tr>
<tr>
<td>Styrene</td>
<td>7.9</td>
<td>0.62</td>
</tr>
<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>3.6</td>
<td>0.27</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>2.6</td>
<td>0.20</td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td>9.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Toluene</td>
<td>3.5</td>
<td>0.38</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>4.2</td>
<td>0.44</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>2.7</td>
<td>0.27</td>
</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
<td>2.6</td>
<td>0.20</td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>2.3</td>
<td>0.19</td>
</tr>
<tr>
<td>Compound</td>
<td>% RSD</td>
<td>MDL (µg/kg)</td>
</tr>
<tr>
<td>---------------------------</td>
<td>-------</td>
<td>-------------</td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>2.7</td>
<td>0.31</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>1.5</td>
<td>0.11</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>4.8</td>
<td>0.45</td>
</tr>
<tr>
<td>m-Xylene/p-Xylene</td>
<td>3.6</td>
<td>0.37</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>3.6</td>
<td>0.33</td>
</tr>
</tbody>
</table>

- **a**: Most compounds spiked at 2 ng/g (2 µg/kg)
- **b**: Incorrect ionization due to methanol
- **c**: Compound detected in unfortified sand at >1 ng
### TABLE 30

**RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery per Replicate (ng)</th>
<th>Mean (ng)</th>
<th>RSD (%)</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 3</td>
<td>RSD (%)</td>
</tr>
<tr>
<td>Benzene</td>
<td>37.6</td>
<td>35.2</td>
<td>38.4</td>
<td>37.1</td>
</tr>
<tr>
<td>Bromochloromethane</td>
<td>20.5</td>
<td>19.4</td>
<td>20.0</td>
<td>20.0</td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>21.1</td>
<td>20.3</td>
<td>22.8</td>
<td>21.4</td>
</tr>
<tr>
<td>Bromoform</td>
<td>23.8</td>
<td>23.9</td>
<td>25.1</td>
<td>24.3</td>
</tr>
<tr>
<td>Bromomethane</td>
<td>21.4</td>
<td>19.5</td>
<td>19.7</td>
<td>20.2</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>27.5</td>
<td>26.6</td>
<td>28.6</td>
<td>27.6</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>25.6</td>
<td>25.4</td>
<td>26.4</td>
<td>25.8</td>
</tr>
<tr>
<td>Chloroethene</td>
<td>25.0</td>
<td>24.4</td>
<td>25.3</td>
<td>24.9</td>
</tr>
<tr>
<td>Chloroform</td>
<td>21.9</td>
<td>20.9</td>
<td>21.7</td>
<td>21.5</td>
</tr>
<tr>
<td>Chloromethane</td>
<td>21.0</td>
<td>19.9</td>
<td>21.3</td>
<td>20.7</td>
</tr>
<tr>
<td>1,2-Dibromo-3-chloropropane</td>
<td>20.8</td>
<td>20.8</td>
<td>21.0</td>
<td>20.9</td>
</tr>
<tr>
<td>1,2-Dibromoethane</td>
<td>20.1</td>
<td>19.5</td>
<td>20.6</td>
<td>20.1</td>
</tr>
<tr>
<td>Dibromomethane</td>
<td>22.2</td>
<td>21.0</td>
<td>22.8</td>
<td>22.0</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>18.0</td>
<td>17.7</td>
<td>17.1</td>
<td>17.6</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>21.2</td>
<td>21.0</td>
<td>20.1</td>
<td>20.8</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>20.1</td>
<td>20.9</td>
<td>19.9</td>
<td>20.3</td>
</tr>
<tr>
<td>Dichlorodifluoromethane</td>
<td>25.3</td>
<td>24.1</td>
<td>25.4</td>
<td>24.9</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>23.0</td>
<td>22.0</td>
<td>22.7</td>
<td>22.6</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>20.6</td>
<td>19.5</td>
<td>19.8</td>
<td>20.0</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>24.8</td>
<td>23.8</td>
<td>24.4</td>
<td>24.3</td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene</td>
<td>21.6</td>
<td>20.0</td>
<td>21.6</td>
<td>21.1</td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td>22.4</td>
<td>21.4</td>
<td>22.2</td>
<td>22.0</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>22.8</td>
<td>22.2</td>
<td>23.4</td>
<td>22.8</td>
</tr>
<tr>
<td>1,1-Dichloropropene</td>
<td>26.3</td>
<td>25.7</td>
<td>28.0</td>
<td>26.7</td>
</tr>
<tr>
<td>cis-1,3-Dichloropropene</td>
<td>20.3</td>
<td>19.5</td>
<td>21.1</td>
<td>20.3</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>24.7</td>
<td>24.5</td>
<td>25.5</td>
<td>24.9</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>23.0</td>
<td>25.3</td>
<td>25.2</td>
<td>24.5</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>26.0</td>
<td>25.7</td>
<td>26.1</td>
<td>25.9</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>13.8</td>
<td>12.7</td>
<td>11.8</td>
<td>12.8</td>
</tr>
<tr>
<td>Styrene</td>
<td>24.2</td>
<td>23.3</td>
<td>23.3</td>
<td>23.6</td>
</tr>
<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>21.4</td>
<td>20.2</td>
<td>21.3</td>
<td>21.0</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>18.6</td>
<td>17.8</td>
<td>19.0</td>
<td>18.5</td>
</tr>
<tr>
<td>Tetrachloroethylene</td>
<td>25.2</td>
<td>24.8</td>
<td>26.4</td>
<td>25.5</td>
</tr>
<tr>
<td>Toluene</td>
<td>28.6</td>
<td>27.9</td>
<td>30.9</td>
<td>29.1</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>15.0</td>
<td>14.4</td>
<td>12.9</td>
<td>14.1</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>28.1</td>
<td>27.2</td>
<td>29.9</td>
<td>28.4</td>
</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
<td>20.8</td>
<td>19.6</td>
<td>21.7</td>
<td>20.7</td>
</tr>
</tbody>
</table>

<sup>a</sup> Statistical analysis not performed.
TABLE 30 (cont.)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Recovery per Replicate (ng)</th>
<th>Mean (ng)</th>
<th>RSD</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 3</td>
<td></td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>26.3</td>
<td>24.9</td>
<td>26.8</td>
<td>26.0</td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>25.9</td>
<td>24.8</td>
<td>26.5</td>
<td>25.7</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>18.8</td>
<td>18.3</td>
<td>19.3</td>
<td>18.8</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>24.8</td>
<td>23.2</td>
<td>23.9</td>
<td>24.0</td>
</tr>
<tr>
<td>m-Xylene/p-Xylene</td>
<td>24.3</td>
<td>23.9</td>
<td>25.3</td>
<td>24.5</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>23.1</td>
<td>22.3</td>
<td>23.4</td>
<td>22.9</td>
</tr>
</tbody>
</table>

a Compound found in unfortified garden soil matrix at >5 ng.
## TABLE 31

**METHOD DETECTION LIMITS AND BOILING POINTS**  
**FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Detection Limit (ng)</th>
<th>Boiling Point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>58</td>
<td>-24</td>
</tr>
<tr>
<td>Bromomethane</td>
<td>26</td>
<td>4</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>14</td>
<td>-13</td>
</tr>
<tr>
<td>Chloroethane</td>
<td>21</td>
<td>13</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>9</td>
<td>40</td>
</tr>
<tr>
<td>Acetone</td>
<td>35</td>
<td>56</td>
</tr>
<tr>
<td>Carbon disulfide</td>
<td>11</td>
<td>46</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>14</td>
<td>32</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>12</td>
<td>57</td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td>11</td>
<td>48</td>
</tr>
<tr>
<td>Chloroform</td>
<td>11</td>
<td>62</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>13</td>
<td>83</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>8</td>
<td>74</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>8</td>
<td>77</td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>11</td>
<td>88</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethene**</td>
<td>23</td>
<td>146</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>12</td>
<td>95</td>
</tr>
<tr>
<td>trans-1,3-Dichloropropene</td>
<td>17</td>
<td>112</td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>11</td>
<td>87</td>
</tr>
<tr>
<td>Dibromochloromethane</td>
<td>21</td>
<td>122</td>
</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
<td>26</td>
<td>114</td>
</tr>
<tr>
<td>Benzene</td>
<td>26</td>
<td>80</td>
</tr>
<tr>
<td>cis-1,3-Dichloropropene</td>
<td>27</td>
<td>112</td>
</tr>
<tr>
<td>Bromoform**</td>
<td>26</td>
<td>150</td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td>11</td>
<td>121</td>
</tr>
<tr>
<td>Toluene</td>
<td>15</td>
<td>111</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>15</td>
<td>132</td>
</tr>
<tr>
<td>Ethylbenzene**</td>
<td>21</td>
<td>136</td>
</tr>
<tr>
<td>Styrene**</td>
<td>46</td>
<td>145</td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>17</td>
<td>24</td>
</tr>
<tr>
<td>Iodomethane</td>
<td>9</td>
<td>43</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>13</td>
<td>78</td>
</tr>
<tr>
<td>Dibromomethane</td>
<td>14</td>
<td>97</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane**</td>
<td>37</td>
<td>157</td>
</tr>
<tr>
<td>total Xylenes**</td>
<td>22</td>
<td>138-144</td>
</tr>
</tbody>
</table>

Footnotes are found on the following page.
* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.

** Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.
### TABLE 32

**VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES
ASSIGNED FOR QUANTITATION (METHOD 5041)**

<table>
<thead>
<tr>
<th>Volatile Internal Standard</th>
<th>Corresponding Analyte</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bromochloromethane</td>
<td>1,4-Difluorobenzene</td>
</tr>
<tr>
<td>Acetone</td>
<td>Benzene</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>Bromodichloromethane</td>
</tr>
<tr>
<td>Bromomethane</td>
<td>Bromoform</td>
</tr>
<tr>
<td>Carbon disulfide</td>
<td>Carbon tetrachloride</td>
</tr>
<tr>
<td>Chloroethane</td>
<td>Chlorodibromomethane</td>
</tr>
<tr>
<td>Chloroform</td>
<td>Dibromomethane</td>
</tr>
<tr>
<td>Chloromethane</td>
<td>1,2-Dichloropropene</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>cis-1,3-Dichloropropene</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>trans-1,3-Dichloropropene</td>
</tr>
<tr>
<td>1,2-Dichloroethane-d₅ (surrogate)</td>
<td>1,1,1-Trichloroethane</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>1,1,2-Trichloroethene</td>
</tr>
<tr>
<td>Trichloroethene</td>
<td></td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td></td>
</tr>
<tr>
<td>Iodomethane</td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td></td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td></td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td></td>
</tr>
<tr>
<td><strong>Chlorobenzene-d₅</strong></td>
<td></td>
</tr>
<tr>
<td>4-Bromofluorobenzene (surrogate)</td>
<td></td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td></td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td></td>
</tr>
<tr>
<td>Styrene</td>
<td></td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td></td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td></td>
</tr>
<tr>
<td>Toluene</td>
<td></td>
</tr>
<tr>
<td>Toluene-d₆ (surrogate)</td>
<td></td>
</tr>
<tr>
<td>1,2,3-Trichloropropene</td>
<td></td>
</tr>
<tr>
<td>Xylenes</td>
<td></td>
</tr>
</tbody>
</table>
# TABLE 33

METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

<table>
<thead>
<tr>
<th>Compound</th>
<th>Boiling Point (°C)</th>
<th>Condensation Point at 20°C (%)</th>
<th>Estimated Detection Limit&lt;sup&gt;a&lt;/sup&gt; (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorodifluoromethane</td>
<td>-30</td>
<td>Gas</td>
<td>0.20</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>-19</td>
<td>Gas</td>
<td>0.11</td>
</tr>
<tr>
<td>1,3-Butadiene</td>
<td>-4</td>
<td>Gas</td>
<td>0.90</td>
</tr>
<tr>
<td>1,2-Dichloro-1,1,2,2-tetrafluoroethane</td>
<td>4</td>
<td>Gas</td>
<td>0.14</td>
</tr>
<tr>
<td>Methyl bromide</td>
<td>4</td>
<td>Gas</td>
<td>0.14</td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>24</td>
<td>88</td>
<td>0.18</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>31</td>
<td>22</td>
<td>0.07</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>40</td>
<td>44</td>
<td>0.05</td>
</tr>
<tr>
<td>1,1,2-Trichloro-trifluoroethane</td>
<td>48</td>
<td>37</td>
<td>0.13</td>
</tr>
<tr>
<td>Chloroform</td>
<td>61</td>
<td>21</td>
<td>0.04</td>
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<td>75</td>
<td>13</td>
<td>0.03</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>77</td>
<td>11</td>
<td>0.03</td>
</tr>
<tr>
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<td>80</td>
<td>10</td>
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<tr>
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<td>87</td>
<td>8</td>
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</tr>
<tr>
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<td>96</td>
<td>5</td>
<td>0.05</td>
</tr>
<tr>
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<td>111</td>
<td>3</td>
<td>0.08</td>
</tr>
<tr>
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<td>121</td>
<td>2</td>
<td>0.03</td>
</tr>
</tbody>
</table>

* Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.
FIGURE 1
GAS CHROMATOGRAM OF VOLATILE ORGANICS

COLUMN: 60 METER x 0.75 MM I.D. VOCOL CAPILLARY

PROGRAM: 10 C FOR 5 MIN., THEN 5 °C/MIN TO 160 C

RETENTION TIME, MIN.
FIGURE 2
GAS CHROMATOGRAM OF VOLATILE ORGANICS
FIGURE 3
GAS CHROMATOGRAM OF VOLATILE ORGANICS
FIGURE 4
GAS CHROMATOGRAM OF TEST MIXTURE

0.5 µg/L PER COMPOUND

1. 1,1-DICHLOROETHYLENE
2. METHYLENE CHLORIDE
3. TRANS-1,2-DICHLOROETHYLENE
4. 1,1 DICHLORETHANE
5. ISOpropylether
6. CHLOROFORM
7. 1,1,1-TRICHLOROETHANE
8. 1,2-DICHLOROETHYLEN
9. CARBON TETRACHLORIDE
10. BENZENE
11. FLUOROBENZENE INT. STD.
12. TRICHLOROETHENE
13. 1,2-DICHLORPROPANE
14. BROMODICHLOOROMETHANE
15. TOLUENE
16. BROMOCHLORPROPANE INT. STD.
17. DIBROMOCHLOROMETHANE
18. TETRACHLOROETHYLENE
19. CHLOROBENZENE
20. ETHERBENZENE
21. 1,3-XYLENE
22. BROMOFORM
23. BROMOBENZENE
24. 1,4-DICHLOROBENZENE
25. 1,2,4-TRICHLOROBENZENE
26. NAPHTHALENE
METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)