

sealed for subsequent analysis. An aliquot can also be poured into a syringe for immediate analysis.

11.5.7.1.2 Purge device compositing - Equal volumes of individual grab samples are added to a purge device to a total volume of 5 or 25 mL. The sample is then analyzed.

11.5.7.1.3 Syringe compositing - In the syringe compositing procedure, equal volumes of individual grab samples are aspirated into a 25 mL syringe while maintaining zero headspace in the syringe. Either the total volume in the syringe or an aliquot is subsequently analyzed. The disadvantage of this technique is that the individual samples must be poured carefully in an attempt to achieve equal volumes of each. An alternate procedure uses multiple 5 mL syringes that are filled with the individual grab samples and then injected sequentially into the 25 mL syringe. 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.

11.5.7.2 Introduce the composited sample into the instrument, using the method of choice. (see Sec. 11.1)

11.5.8 Add appropriate volumes of the surrogate spiking solution and the internal standard spiking solution to each sample either manually or by autosampler to achieve the desired concentrations. The surrogate and internal standards may be mixed and added as a single spiking solution.

If a more sensitive mass spectrometer is employed to achieve lower quantitation levels, more dilute surrogate and internal standard solutions may be required.

11.5.9 Add 10 μ L of the matrix spike solution (Sec. 7.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 μ g/L of each matrix spike standard.

11.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 9.5 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

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

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

11.5.10.1 For direct injection, inject 1 to 2 μ L 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).

11.5.10.2 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.

11.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the upper limit of 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.

11.5.11.1 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. Depending on the extent of the decontamination procedures, recalibration may be necessary.

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

11.5.12 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below:

$$\text{Dwell Time for the Group} = \frac{\text{Laboratory's Scan Time (msec)}}{\text{Total Ions in the Group}}$$

11.6 Analyte identification

11.6.1 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.

11.6.1.1 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.

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

11.6.1.3 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%.)

11.6.1.4 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 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported.

11.6.1.5 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.

11.6.1.6 Examination of extracted ion current profiles (EICP) 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.

11.6.2 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 $\pm 20\%$. (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.

11.7 Quantitation

11.7.1 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.

11.7.1.1 It is highly recommended to use the integration produced by the software if the integration is correct because the software should produce more consistent integrations. However, manual integrations are necessary when the software does not produce proper integrations due to improper baseline selection, the correct peak is missed, a coelution is integrated, a peak is partially integrated, etc. The analyst is responsible for ensuring that the integration is correct whether performed by the software or done manually.

11.7.1.2 Manual integrations should not be substituted for proper maintenance of the instrument or setup of the method (e.g. retention time updates, integration parameter files, etc). The analyst should seek to minimize manual integration by properly maintaining the instrument, updating retention times, and configuring peak integration parameters.

11.7.2 If the RSD of a compound's response factor is 20% or less, then the concentration in the extract may be determined using the average response factor (\overline{RF}) from initial calibration data (Sec. 11.3.5). See Method 8000 for the equations describing internal standard calibration and either linear or non-linear calibrations.

11.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 11.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The resulting concentration should

be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences.

11.7.4 Structural isomers that produce very similar mass spectra should be quantitated 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 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported.

11.7.5 Quantitation of multicomponent parameters such as gasoline range organics (GROs) and total petroleum hydrocarbons (TPH) using the Method 8260 recommended internal standard quantitation technique is beyond the scope of this method. Typically, analyses for these parameters are performed using GC/FID or GC with a MS detector capability that is available with Method 8015.

12.0 DATA ANALYSIS AND CALCULATIONS

See Sec. 11.7 for information on data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 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 example lower limits of quantitation are presented in Table 1.

13.3 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. Example lower limits of quantitation were also calculated from these data and are presented in Table 2.

13.4 Initial demonstration of capability data from two EPA Regional laboratories were submitted using purge-and-trap (Method 5030) with water spiked at 20 µg/L and analyzed on a narrow-bore column. The accuracy and precision data for these studies are presented in Tables 8 and 9.

13.5 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 12 and 13 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.

13.6 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 waste landfill, and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 20 µg/kg. These data are listed in Tables 18, 19, and 20. 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%.

13.6.1 In general, the recoveries of the analytes from the sand matrix are the highest, the hazardous waste landfill 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.

13.6.2 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.

13.7 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 14 to 17.

13.8 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 21 to 25.

13.9 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in a garden soil matrix. Replicate samples were fortified with the analytes at a concentration of 20 µg/kg. These data are listed in Table 28. All data were calculated using the internal standards listed for each analyte in Table 26. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

13.9.1 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 benzene, toluene, and xylenes. This is due to contamination of the soil prior to sample collection.

13.9.2 The example lower limits of quantitation using Method 5021 are listed in Table 27 and 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 lower limits of quantitation were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

13.10 The lower limits of quantitation listed in Table 29 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 lower limit of quantitation 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 lower limit of quantitation for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

13.11 The lower limit of quantitation for samples 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 31). Matrix effects may cause the individual compound quantitation limits to be higher.

13.12 The recommended internal standards with corresponding analytes assigned for quantitation that are appropriate for Method 5041 are listed in Table 30.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

EXAMPLE CHROMATOGRAPHIC RETENTION TIMES AND LOWER LIMITS OF QUANTITATION FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			LOQ ^d (µg/L)
	Column 2A ^a	Column 2B ^b	Column 2C ^c	
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			LOQ ^d (µg/L)
	Column 1A ^a	Column 2B ^b	Column 2C ^c	
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03

TABLE 1 (cont.)

Compound	Retention Time (minutes)			LOQ ^d (µg/L)
	Column 1A ^a	Column 2B ^b	Column 2C ^c	
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene- <i>d</i> ₅	23.10			
1,4-Dichlorobenzene- <i>d</i> ₄	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene- <i>d</i> ₄	32.30	19.08	27.25	
Dichloroethane- <i>d</i> ₄	12.08			
Dibromofluoromethane	--			
Toluene- <i>d</i> ₈	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

^a Column 2A - 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 2B - 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 2C - 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 Limit of quantitation based on a 25-mL sample volume.

TABLE 2

EXAMPLE CHROMATOGRAPHIC RETENTION TIMES AND LOWER LIMITS OF
QUANTITATION FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY
COLUMNS

Compound	Retention Time (minutes)	
	Column 4 ^a	Lower Limit of Quantitation (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes)	Lower Limit of Quantitation (µg/L)
	Column 4 ^a	
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

^a Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

^b Lower limit of quantitation based on a 25-mL sample volume.

TABLE 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA^a

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

^a The criteria in this table are intended to be used as default criteria for quadrupole instrumentation if optimized manufacturer's operating conditions are not available. Alternate tuning criteria may be employed (e.g., CLP or Method 524.2), provided that method performance is not adversely affected. See Sec. 11.3.1

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

^a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

^b Data provided by EPA Region III laboratory.

TABLE 5

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d ₄	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
β -Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene- d_6	84	83
Bromobenzene- d_5	82	162
Bromochloromethane- d_2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene- d_5	117	
1,4-Dichlorobenzene- d_4	152	115, 150
1,1,2-Trichloroethane- d_3	100	
4-Bromofluorobenzene	95	174, 176

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Chloroform- d_1	84	
Dibromofluoromethane	113	
Dichloroethane- d_4	102	
Toluene- d_8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery ^a	Standard Deviation of Recovery ^b	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery ^a	Standard Deviation of Recovery ^b	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

^a Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

^b Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery ^a	Standard Deviation of Recovery ^b	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery ^a	Standard Deviation of Recovery ^b	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

^a Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

US EPA REGION III LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Replicate #1	Replicate #2	Replicate #3	Replicate #4	Average	%Rec	RSD
Acetone	20	23.78	21.41	21.10	21.95	22.06	110.3	5.4
Benzene	20	19.42	19.31	18.41	20.08	19.31	96.5	3.6
Bromobenzene	20	22.74	19.32	18.61	19.52	20.05	100.2	9.2
Bromochloromethane	20	22.87	19.58	18.91	19.60	20.24	101.2	8.8
Bromodichloromethane	20	18.07	18.10	17.57	18.69	18.11	90.5	2.5
Bromoform	20	18.97	19.39	18.88	19.09	19.08	95.4	1.2
Bromomethane	20	18.93	18.76	17.77	19.86	18.83	94.2	4.5
2-Butanone	20	21.81	20.70	21.61	22.01	21.53	107.7	2.7
n-Butylbenzene	20	22.16	19.14	18.04	19.12	19.62	98.1	9.0
sec-Butylbenzene	20	22.84	19.36	18.15	19.39	19.94	99.7	10.1
tert-Butylbenzene	20	21.87	18.62	17.64	18.62	19.19	95.9	9.6
Carbon disulfide	20	19.01	18.69	17.25	20.23	18.80	94.0	6.5
Carbon tetrachloride	20	19.46	18.74	17.77	20.34	19.08	95.4	5.7
Chlorobenzene	20	19.54	19.39	18.77	20.23	19.48	97.4	3.1
Chloroethane	20	18.86	18.89	17.06	19.95	18.69	93.5	6.4
2-Chloroethylvinyl ether	20	18.26	16.54	16.31	16.51	16.91	84.5	5.4
Chloroform	20	19.47	19.62	18.60	20.14	19.46	97.3	3.3
Chloromethane	20	18.89	18.27	16.78	19.37	18.33	91.6	6.1
2-Chlorotoluene	20	22.82	19.45	18.52	19.69	20.12	100.6	9.3
4-Chlorotoluene	20	22.46	19.08	18.19	19.38	19.78	98.9	9.4
Cyclohexane	20	19.10	18.60	17.46	20.20	18.84	94.2	6.0
1,2-Dibromo-3-chloropropane	20	18.90	18.07	18.54	18.64	18.54	92.7	1.9

TABLE 8 (cont.)

US EPA REGION III LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Replicate #1	Replicate #2	Replicate #3	Replicate #4	Average	%Rec	RSD
Dibromochloromethane	20	19.70	19.62	19.23	19.83	19.60	98.0	1.3
1,2-Dibromoethane	20	19.95	19.92	19.83	20.39	20.02	100.1	1.2
Dibromomethane	20	22.77	19.72	19.47	20.21	20.54	102.7	7.4
1,2-Dichlorobenzene	20	18.85	18.89	18.29	19.56	18.90	94.5	2.8
1,3-Dichlorobenzene	20	19.27	19.22	18.71	19.91	19.28	96.4	2.6
1,4-Dichlorobenzene	20	19.60	19.49	18.81	20.20	19.53	97.6	2.9
Dichlorodifluoromethane	20	21.07	20.36	18.76	22.20	20.60	103.0	7.0
1,1-Dichloroethane	20	19.23	19.42	18.22	20.15	19.26	96.3	4.1
1,2-Dichloroethane	20	20.15	20.21	19.71	20.67	20.19	100.9	1.9
1,1-Dichloroethene	20	19.36	18.98	17.78	20.59	19.18	95.9	6.0
cis-1,2-Dichloroethene	20	18.56	18.67	17.77	19.40	18.60	93.0	3.6
trans-1,2-Dichloroethene	20	19.26	18.96	17.85	20.04	19.03	95.1	4.8
1,2-Dichloropropane	20	19.73	19.46	18.74	20.18	19.53	97.6	3.1
1,3-Dichloropropane	20	22.23	19.68	19.51	19.59	20.25	101.3	6.5
2,2-Dichloropropane	20	23.16	19.40	17.71	19.08	19.84	99.2	11.8
1,1-Dichloro-1-propene	20	23.24	19.70	18.29	19.97	20.30	101.5	10.3
cis-1,3-Dichloropropene	20	18.96	19.09	18.47	19.54	19.02	95.1	2.3
trans-1,3-Dichloropropene	20	20.19	20.31	19.57	20.36	20.11	100.5	1.8
Ethylbenzene	20	19.55	19.28	18.21	20.21	19.31	96.6	4.3
Hexachlorobutadiene	20	21.14	18.20	17.30	18.34	18.75	93.7	8.9
2-Hexanone	20	23.39	21.20	21.24	22.01	21.96	109.8	4.7
Isopropylbenzene	20	19.13	18.91	17.81	19.84	18.92	94.6	4.4

TABLE 8 (cont.)

US EPA REGION III LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Replicate #1	Replicate #2	Replicate #3	Replicate #4	Average	%Rec	RSD
p-Isopropyltoluene	20	22.28	18.98	17.93	18.93	19.53	97.7	9.7
Methylene chloride	20	20.03	19.89	20.17	20.56	20.16	100.8	1.4
Methyl acetate	20	20.21	19.73	20.34	20.77	20.26	101.3	2.1
Methyl cyclohexane	20	19.24	18.80	17.75	20.18	18.99	95.0	5.3
4-Methyl-2-pentanone	20	22.00	21.19	21.44	22.00	21.66	108.3	1.9
Methyl tert-butyl ether	20	20.23	20.30	19.82	20.41	20.19	101.0	1.3
Naphthalene	20	20.98	18.60	18.42	18.89	19.22	96.1	6.2
n-Propylbenzene	20	22.56	19.20	18.05	19.31	19.78	98.9	9.8
Styrene	20	16.72	16.46	15.90	16.87	16.49	82.4	2.6
1,1,1,2-Tetrachloroethane	20	22.67	19.12	18.58	19.46	19.96	99.8	9.2
1,1,2,2-Tetrachloroethane	20	20.54	20.21	20.24	20.96	20.49	102.4	1.7
Tetrachloroethene	20	18.88	18.57	17.32	19.67	18.61	93.1	5.2
Toluene	20	20.01	19.59	18.78	20.51	19.72	98.6	3.7
1,2,3-Trichlorobenzene	20	21.68	19.12	18.59	19.20	19.65	98.2	7.0
1,2,4-Trichlorobenzene	20	19.39	18.50	18.13	19.04	18.77	93.8	3.0
1,1,1-Trichloroethane	20	19.06	18.58	17.47	19.95	18.77	93.8	5.5
1,1,2-Trichloroethane	20	19.76	19.91	19.47	20.33	19.87	99.3	1.8
Trichloroethene	20	20.19	19.84	18.42	20.67	19.78	98.9	4.9
Trichlorofluoromethane	20	18.25	17.98	16.79	18.98	18.00	90.0	5.1
1,2,3-Trichloropropane	20	21.83	19.89	19.76	20.32	20.45	102.3	4.6

TABLE 8 (cont.)

US EPA REGION III LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Replicate #1	Replicate #2	Replicate #3	Replicate #4	Average	%Rec	RSD
1,1,2-Trichloro-1,2,2-trifluoroethane	20	20.33	19.65	18.54	21.18	19.93	99.6	5.6
1,2,4-Trimethylbenzene	20	22.67	19.41	18.47	19.45	20.00	100.0	9.2
1,3,5-Trimethylbenzene	20	22.51	19.13	18.09	19.20	19.73	98.7	9.7
Vinyl acetate	20	22.23	19.92	19.44	19.86	20.36	101.8	6.2
Vinyl chloride	20	19.85	19.72	18.08	21.12	19.69	98.5	6.3
o-Xylene	20	19.78	19.63	18.87	20.46	19.69	98.4	3.3
m & p-Xylene	40	39.08	38.42	36.45	40.21	38.54	96.4	4.1
Surrogates								
1,2-Dichloroethane- <i>d</i> ₄	20	20.33	20.33	20.35	20.41	20.36	101.8	0.2
Toluene- <i>d</i> ₈	20	20.39	20.24	20.36	20.21	20.30	101.5	0.4
Bromofluorobenzene	20	20.18	20.17	20.12	20.19	20.17	100.8	0.2

TABLE 9

US EPA REGION VI LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Average	%Rec	RSD
Acetone	20	18.69	22.81	19.57	21.73	21.30	20.82	104	8.0
Benzene	20	17.99	19.64	18.25	19.77	20.06	19.14	96	5.0
Bromodichloromethane	20	17.86	20.40	18.39	20.45	20.02	19.42	97	6.2
Bromoform	20	18.47	20.16	18.12	20.83	20.39	19.59	98	6.2
Bromomethane	20	18.53	21.27	19.25	20.66	21.31	20.20	101	6.2
2-Butanone	20	17.20	20.88	18.33	20.86	20.84	19.62	98	8.9
Carbon disulfide	20	18.63	20.86	19.00	20.99	21.50	20.20	101	6.4
Carbon tetrachloride	20	17.82	20.11	18.41	19.79	20.19	19.26	96	5.6
Chlorobenzene	20	18.11	20.65	18.47	20.60	20.63	19.69	98	6.5
Chloroethane	20	21.84	20.31	19.18	20.57	21.70	20.72	104	5.3
Chloroform	20	18.48	21.32	19.10	21.28	20.44	20.12	101	6.4
Chloromethane	20	18.35	20.08	19.11	20.38	19.18	19.42	97	4.2
Cyclohexane	20	18.00	20.07	18.01	19.55	20.35	19.20	96	5.9
1,2-Dibromo-3-chloropropane	20	17.23	20.88	18.03	21.62	20.69	19.69	98	9.8
Dibromochloromethane	20	17.36	20.07	18.23	19.76	20.07	19.10	95	6.5
1,2-Dibromoethane	20	17.97	20.06	18.13	20.01	20.88	19.41	97	6.6
1,2-Dichlorobenzene	20	17.74	19.92	18.11	19.41	20.05	19.05	95	5.6
1,3-Dichlorobenzene	20	17.95	20.10	17.98	19.90	20.25	19.24	96	6.1
1,4-Dichlorobenzene	20	18.05	19.66	18.22	19.47	19.67	19.01	95	4.3
Dichlorodifluoromethane	20	18.81	21.17	19.46	20.98	20.76	20.24	101	5.1
1,1-Dichloroethane	20	18.34	20.86	18.57	20.43	20.69	19.78	99	6.2
1,2-Dichloroethane	20	18.94	21.32	19.35	21.44	20.63	20.34	102	5.6

TABLE 9 (cont.)

US EPA REGION VI LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep #5	Average	%Rec	RSD
1,1-Dichloroethene	20	17.69	20.18	18.44	21.12	19.81	19.45	97	7.1
cis-1,2-Dichloroethene	20	18.23	20.74	18.49	20.13	19.60	19.44	97	5.5
trans-1,2-Dichloroethene	20	18.29	20.50	18.25	19.89	20.04	19.39	97	5.4
1,2-Dichloropropane	20	17.97	20.35	18.23	19.98	20.44	19.39	97	6.2
cis-1,3-Dichloropropene	20	16.29	17.93	16.14	17.93	17.77	17.21	86	5.3
trans-1,3-Dichloropropene	20	16.83	18.88	16.93	18.46	18.50	17.92	90	5.4
Ethylbenzene	20	17.03	19.05	17.20	18.82	19.04	18.23	91	5.6
2-Hexanone	20	17.00	20.59	18.63	19.59	21.16	19.39	97	8.5
Isopropylbenzene	20	17.22	19.51	17.32	18.75	19.19	18.40	92	5.8
Methylene chloride	20	18.56	20.23	18.76	20.53	20.06	19.63	98	4.6
Methyl acetate	20	18.74	21.20	19.04	21.21	20.57	20.15	101	5.9
Methyl cyclohexane	20	18.36	20.51	18.33	20.50	20.26	19.59	98	5.8
4-Methyl-2-pentanone	20	15.91	19.04	16.86	18.10	18.86	17.75	89	7.6
Methyl tert-butyl ether	20	17.52	20.14	18.03	20.41	19.46	19.11	96	6.7
Styrene	20	17.59	19.93	17.88	19.52	19.87	18.96	95	6.0
1,1,2,2-Tetrachloroethane	20	18.29	21.59	19.00	22.17	21.09	20.43	102	8.3
Tetrachloroethene	20	18.12	20.53	18.80	20.79	20.91	19.83	99	6.5
Toluene	20	18.36	20.57	18.77	20.25	20.85	19.76	99	5.7
1,2,4-Trichlorobenzene	20	16.97	18.95	17.09	18.38	18.86	18.05	90	5.3
1,1,1-Trichloroethane	20	18.30	19.87	18.49	20.37	19.91	19.39	97	4.8
1,1,2-Trichloroethane	20	17.68	19.74	18.02	20.37	19.84	19.13	96	6.3
Trichloroethene	20	17.89	19.49	18.20	19.80	20.06	19.09	95	5.1

TABLE 9 (cont.)

US EPA REGION VI LABORATORY INITIAL DEMONSTRATION OF CAPABILITY DATA FOR
PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED
WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Spike Conc. (µg/L)	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Average	%Rec	RSD
Trichlorofluoromethane	20	19.53	21.52	19.86	21.76	21.68	20.87	104	5.2
1,1,2-Trichloro-1,2,2-trifluoroethane	20	19.48	21.10	18.18	21.16	21.46	20.28	101	6.9
Vinyl chloride	20	19.22	21.48	19.35	21.33	20.97	20.47	102	5.4
o-Xylene	20	17.89	19.87	17.58	19.38	20.00	18.94	95	6.0
m & p-Xylene	40	36.15	40.29	36.51	39.44	40.57	38.59	96	5.5
Surrogates									
1,2-Dichloroethane- <i>d</i> ₄	50	45.62	45.78	45.94	46.31	45.35	45.80	92	0.8
Toluene- <i>d</i> ₈	50	47.00	47.17	47.65	47.24	46.93	47.20	94	0.6
Bromofluorobenzene	50	48.40	49.46	49.32	48.61	49.48	49.05	98	1.0

TABLE 10

EXAMPLE SURROGATE SPIKE RECOVERY LIMITS
FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene ^a	86-115	74-121
Dibromofluoromethane ^a	86-118	80-120
Toluene- <i>d</i> ₈ ^a	88-110	81-117
Dichloroethane- <i>d</i> ₄ ^a	80-120	80-120

^a Single laboratory data, for guidance only.

TABLE 11

RECOMMENDED QUANTITY OF EXTRACT FOR ANALYSIS OF
HIGH CONCENTRATION SAMPLES

Approximate Concentration Range (µg/kg)	Volume of Extract ^a
500 - 10,000	100 µL
1,000 - 20,000	50 µL
5,000 - 100,000	10 µL
25,000 - 500,000	100 µL of 1/50 dilution ^b

Calculate appropriate dilution factor for concentrations exceeding this table.

^a 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.

^b Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

TABLE 12

DIRECT INJECTION ANALYSIS OF NEW OIL AT VARIOUS CONCENTRATIONS
(METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

* Alternate mass employed

** IS quantitation

Data are taken from Reference 9.

TABLE 13

SINGLE LABORATORY PERFORMANCE
DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene- <i>d</i> ₆	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*, **	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride- ¹³ C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene- <i>d</i> ₅	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform- <i>d</i> ₁	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene- <i>d</i> ₄	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene- <i>d</i> ₂	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*, **	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene- <i>d</i> ₁₀	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane- ¹³ C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene- <i>d</i> ₅	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine- <i>d</i> ₅	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl ₃ F ₃ ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene- <i>d</i> ₈	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene- <i>d</i> ₁	55	44	2.8	0.5

TABLE 13 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene- <i>d</i> ₁₀	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene- <i>d</i> ₁₀	67	44	3.7	10.0

* Alternate mass employed

** IS quantitation

ND = Not Detected

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

TABLE 14

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

Target Compound	Surrogate	Internal Standard
Acetone	<i>d</i> ₆ -Acetone	<i>d</i> ₈ -Isopropyl alcohol
Acetonitrile	<i>d</i> ₃ -Acetonitrile	<i>d</i> ₈ -Isopropyl alcohol
Acrylonitrile	<i>d</i> ₈ -Isopropyl alcohol	
Allyl alcohol	<i>d</i> ₇ -Dimethyl formamide	
Crotonaldehyde	<i>d</i> ₈ -Isopropyl alcohol	
1,4-Dioxane	<i>d</i> ₈ -1,4-Dioxane	<i>d</i> ₇ -Dimethyl formamide
Isobutyl alcohol	<i>d</i> ₇ -Dimethyl formamide	
Methanol	<i>d</i> ₃ -Methanol	<i>d</i> ₈ -Isopropyl alcohol
Methyl ethyl ketone	<i>d</i> ₈ -Isopropyl alcohol	
N-Nitroso-di-n-butylamine	<i>d</i> ₇ -Dimethyl formamide	
Paraldehyde	<i>d</i> ₇ -Dimethyl formamide	
2-Picoline	<i>d</i> ₇ -Dimethyl formamide	
Propionitrile	<i>d</i> ₈ -Isopropyl alcohol	
Pyridine	<i>d</i> ₅ -Pyridine	<i>d</i> ₇ -Dimethyl formamide
o-Toluidine	<i>d</i> ₇ -Dimethyl formamide	

TABLE 15

RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/ μ L)
Internal Standards	
<i>d</i> ₅ -benzyl alcohol	10.0
<i>d</i> _{1,4} -Diglyme	10.0
<i>d</i> ₇ -Dimethyl formamide	10.0
<i>d</i> ₈ -Isopropyl alcohol	10.0
Surrogates	
<i>d</i> ₆ -Acetone	10.0
<i>d</i> ₃ -Acetonitrile	10.0
<i>d</i> ₈ -1,4-Dioxane	10.0
<i>d</i> ₃ -Methanol	10.0
<i>d</i> ₅ -Pyridine	10.0
Target Compounds	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 16

CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion ^a	Secondary Ions	Retention Time (min) ^b
Internal Standards			
<i>d</i> ₈ -Isopropyl alcohol	49		1.75
<i>d</i> ₁₄ -Diglyme	66	98,64	9.07
<i>d</i> ₇ -Dimethyl formamide	50	80	9.20
Surrogates			
<i>d</i> ₆ -Acetone	46	64,42	1.03
<i>d</i> ₃ -Methanol	33	35,30	1.75
<i>d</i> ₃ -Acetonitrile	44	42	2.63
<i>d</i> ₈ -1,4-Dioxane	96	64,34	3.97
<i>d</i> ₅ -Pyridine	84	56,79	6.73
<i>d</i> ₅ -Phenol ^c	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile ^c	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone ^c	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline ^c	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol ^c	94	66,65	15.43

^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 17

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

Compound	25 ppb Spike		100 ppb Spike		500 ppb Spike	
	Mean %R	%RSD	Mean %R	%RSD	Mean %R	%RSD
<i>d</i> ₆ -Acetone	66	24	69	14	65	16
<i>d</i> ₃ -Acetonitrile	89	18	80	18	70	10
<i>d</i> ₈ -1,4-Dioxane	56	34	58	11	61	18
<i>d</i> ₃ -Methanol	43	29	48	19	56	14
<i>d</i> ₅ -Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di- n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

^a Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrupole mass spectrometer in the selected ion monitoring mode.

TABLE 18

RECOVERIES IN SAND SAMPLES FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 19
RECOVERIES IN HAZARDOUS WASTE LANDFILL SOILS FORTIFIED AT 20 µg/kg (ANALYSIS
BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2

TABLE 20
RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5035)

Compound	Recovery per Replicate (ng)					Mean	RSD	Mean Rec
	1	2	3	4	5			
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 21

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL
USING VACUUM DISTILLATION (METHOD 5032)^a

Compound	Soil/H ₂ O ^b Recovery		Soil/Oil ^c Recovery		Soil/Oil/H ₂ O ^d Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT ^e	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT ^f	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 21 (cont.)

Compound	Soil/H ₂ O ^b Recovery		Soil/Oil ^c Recovery		Soil/Oil/H ₂ O Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d ₈	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

^a 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 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 22

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

Compound	Efficiency	
	Mean (%)	RSD (%)
Chloromethane	N/A ^b	
Bromomethane	N/A ^b	
Vinyl chloride	N/A ^b	
Chloroethane	N/A ^b	
Methylene chloride	CONT ^c	
Acetone	CONT ^c	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT ^d	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT ^d	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT ^d	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT ^c	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 22 (cont.)

Compound	Efficiency	
	Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d ₈	88	24
Bromofluorobenzene	52	15

- ^a 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.
- ^b No analyses.
- ^c Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.
- ^d Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER
USING VACUUM DISTILLATION (METHOD 5032)^a

Compound	5 mL H ₂ O Recovery		20 mL H ₂ O Recovery		20 mL H ₂ O/Oil ^b Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT ^c	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14

TABLE 23 (cont.)

Compound	5 mL H ₂ O ^b Recovery		20 mL H ₂ O ^c Recovery		20 mL H ₂ O/Oil Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d ₈	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

^a 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.

^b 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.

^c Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

VOLATILE ORGANIC ANALYTE RECOVERY FROM FISH OIL
USING VACUUM DISTILLATION (METHOD 5032)^a

Compound	Recovery	
	Mean (%)	RSD (%)
Chloromethane	N/A ^b	
Bromomethane	N/A ^b	
Vinyl chloride	N/A ^b	
Chloroethane	N/A ^b	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT ^c	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT ^c	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT ^c	
4-Methyl-2-pentanone	INT ^c	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 24 (cont.)

Compound	Recovery	
	Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane-d ₄	137	30
Toluene-d ₈	84	6
Bromofluorobenzene	48	2

^a Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish oil 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.

^b Not analyzed.

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

TABLE 25

EXAMPLE LOWER LIMITS OF QUANTITATION FOR VOLATILE ORGANIC ANALYTES
IN FISH OIL (METHOD 5032)^a

Compound	Lower Limit of Quantitation (ppb)	
	External Standard Method	Internal Standard Method
Chloromethane	N/A ^b	N/A ^b
Bromomethane	N/A ^b	N/A ^b
Vinyl chloride	N/A ^b	N/A ^b
Chloroethane	N/A ^b	N/A ^b
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT ^c	INT ^c
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT ^c	INT ^c
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT ^c	INT ^c
4-Methyl-2-pentanone	INT ^c	INT ^c
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70

TABLE 25 (cont.)

- ^a Method quantitation limits (MQLs) are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MQLs 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. MQLs can be used to establish the lower limit of instrument quantitation, however, since they are statistical approximations of the actual method sensitivity, it is recommended that the lowest calibration concentration be used to establish the minimum quantitation limit.
- ^b No analyses.
- ^c Interference by co-eluting compounds prevented accurate quantitation.

TABLE 26

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS
(METHOD 5021)

Chloroform-d ₁	1,1,2-TCA-d ₃	Bromobenzene-d ₅
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 27

EXAMPLE PRECISION AND STATISTICAL MINIMUM QUANTITATION LIMIT (MQL)
DETERMINED FOR ANALYSIS OF FORTIFIED SAND^a (METHOD 5021)

Compound	% RSD	MQL (µg/kg)
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5 ^b
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62 ^c
Naphthalene	16.8	3.4 ^c
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2 ^c
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 27 (cont.)

Compound	% RSD	MQL ($\mu\text{g}/\text{kg}$)
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

- ^a Most compounds spiked at 2 ng/g (2 $\mu\text{g}/\text{kg}$)
^b Incorrect ionization due to methanol
^c Compound detected in unfortified sand at >1 ng

TABLE 28

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

Compound	Recovery per Replicate (ng)			Mean (ng)	RSD	Recovery (%)
	Sample 1	Sample 2	Sample 3			
Benzene	37.6	35.2	38.4	37.1	3.7	185 ^a
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104 ^a
1,2-Dibromo-3-chloro- propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130 ^a
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8 ^a
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146 ^a
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 28 (cont.)

Compound	Recovery per Replicate (ng)			Mean (ng)	RSD	Recovery (%)
	Sample 1	Sample 2	Sample 3			
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

^a Compound found in unfortified garden soil matrix at >5 ng.

TABLE 29

EXAMPLE MINIMUM QUANTITATION LIMITS (MQL) AND BOILING POINTS
FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)^a

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

Footnotes are found on the following page.

TABLE 29 (cont.)

- * The method quantitation limit (MQL) is defined in Chapter One. The quantitation 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. MQLs can be used to establish the lower limit of instrument quantitation, however, since they are statistical approximations of the actual method sensitivity, it is recommended that the lowest calibration concentration be used to establish the minimum quantitation limit.

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

TABLE 30

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

Bromochloromethane

Acetone
Acrylonitrile
Bromomethane
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane-d₄ (surrogate)
1,1-Dichloroethene
Trichloroethene
trans-1,2-Dichloroethene
Iodomethane
Methylene chloride
Trichlorofluoromethane
Vinyl chloride

Chlorobenzene-d₅

4-Bromofluorobenzene (surrogate)
Chlorobenzene
Ethylbenzene
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d₈ (surrogate)
1,2,3-Trichloropropane
Xylenes

1,4-Difluorobenzene

Benzene
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorodibromomethane
Dibromomethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane

TABLE 31

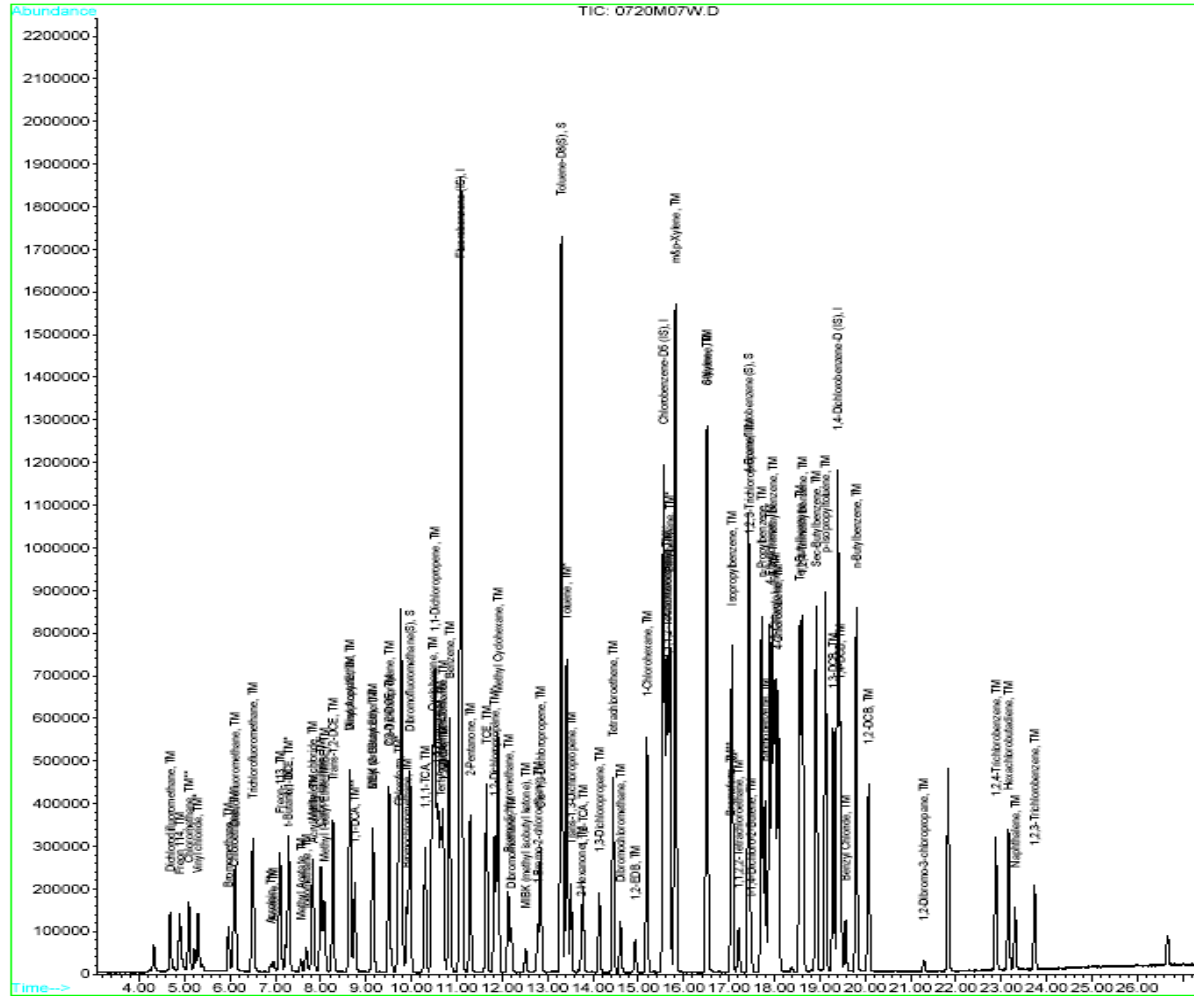
METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Quantitation Limit ^a (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

^a Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack quantitation limits.

FIGURE 1
 EXAMPLE GAS CHROMATOGRAM OF VOLATILE ORGANICS
 (Provided Courtesy APPL, Inc.)

File : M:\MAX\DATA\M050720\0720M07W.D
 Operator : RP
 Acquired : 20 Jul 05 19:50 using AcqMethod PM82602
 Instrument : Max
 Sample Name: Vol Std 07-20-05Z@10ug/L
 Misc Info : Water 10mL w/IS: 07-12-05AQ
 Vial Number: 7



8260C- 91

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 August 2006

FIGURE 2
EXAMPLE GAS CHROMATOGRAM OF VOLATILE ORGANICS
(Provided Courtesy EPA Region III)

