

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

<sup>a</sup> 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.

<sup>b</sup> 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.

<sup>c</sup> 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.

<sup>d</sup> 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 <sup>a</sup>	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 <sup>a</sup>	
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

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

<sup>b</sup> Lower limit of quantitation based on a 25-mL sample volume.

TABLE 4

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

Volatile Compounds	Minimum Response Factor (RF) <sup>a</sup>	Typical Response Factor (RF) <sup>b</sup>
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) <sup>a</sup>	Typical Response Factor (RF) <sup>b</sup>
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

<sup>a</sup> 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.

<sup>b</sup> Data provided by EPA Region III laboratory.

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 <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	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 <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	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

<sup>a</sup> Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

<sup>b</sup> 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 <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	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 <sup>a</sup>	Standard Deviation of Recovery <sup>b</sup>	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

<sup>a</sup> 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> <sub>4</sub>	20	20.33	20.33	20.35	20.41	20.36	101.8	0.2
Toluene- <i>d</i> <sub>8</sub>	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> <sub>4</sub>	50	45.62	45.78	45.94	46.31	45.35	45.80	92	0.8
Toluene- <i>d</i> <sub>8</sub>	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 <sup>a</sup>	86-115	74-121
Dibromofluoromethane <sup>a</sup>	86-118	80-120
Toluene- <i>d</i> <sub>8</sub> <sup>a</sup>	88-110	81-117
Dichloroethane- <i>d</i> <sub>4</sub> <sup>a</sup>	80-120	80-120

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

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> <sub>6</sub>	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- <sup>13</sup> C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene- <i>d</i> <sub>5</sub>	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform- <i>d</i> <sub>1</sub>	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene- <i>d</i> <sub>4</sub>	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> <sub>2</sub>	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> <sub>10</sub>	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane- <sup>13</sup> 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> <sub>5</sub>	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine- <i>d</i> <sub>5</sub>	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl <sub>3</sub> F <sub>3</sub> ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene- <i>d</i> <sub>8</sub>	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene- <i>d</i> <sub>1</sub>	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> <sub>10</sub>	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene- <i>d</i> <sub>10</sub>	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 15

## RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/ $\mu$ L)
Internal Standards	
<i>d</i> <sub>5</sub> -benzyl alcohol	10.0
<i>d</i> <sub>14</sub> -Diglyme	10.0
<i>d</i> <sub>7</sub> -Dimethyl formamide	10.0
<i>d</i> <sub>8</sub> -Isopropyl alcohol	10.0
Surrogates	
<i>d</i> <sub>6</sub> -Acetone	10.0
<i>d</i> <sub>3</sub> -Acetonitrile	10.0
<i>d</i> <sub>8</sub> -1,4-Dioxane	10.0
<i>d</i> <sub>3</sub> -Methanol	10.0
<i>d</i> <sub>5</sub> -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 <sup>a</sup>	Secondary Ions	Retention Time (min) <sup>b</sup>
Internal Standards			
<i>d</i> <sub>8</sub> -Isopropyl alcohol	49		1.75
<i>d</i> <sub>14</sub> -Diglyme	66	98,64	9.07
<i>d</i> <sub>7</sub> -Dimethyl formamide	50	80	9.20
Surrogates			
<i>d</i> <sub>6</sub> -Acetone	46	64,42	1.03
<i>d</i> <sub>3</sub> -Methanol	33	35,30	1.75
<i>d</i> <sub>3</sub> -Acetonitrile	44	42	2.63
<i>d</i> <sub>8</sub> -1,4-Dioxane	96	64,34	3.97
<i>d</i> <sub>5</sub> -Pyridine	84	56,79	6.73
<i>d</i> <sub>5</sub> -Phenol <sup>c</sup>	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 <sup>c</sup>	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone <sup>c</sup>	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 <sup>c</sup>	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol <sup>c</sup>	94	66,65	15.43

<sup>a</sup> These ions were used for quantitation in selected ion monitoring.

<sup>b</sup> 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.

<sup>c</sup> 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<sup>a</sup> (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> <sub>6</sub> -Acetone	66	24	69	14	65	16
<i>d</i> <sub>3</sub> -Acetonitrile	89	18	80	18	70	10
<i>d</i> <sub>8</sub> -1,4-Dioxane	56	34	58	11	61	18
<i>d</i> <sub>3</sub> -Methanol	43	29	48	19	56	14
<i>d</i> <sub>5</sub> -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

<sup>a</sup> 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)<sup>a</sup>

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

<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 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.

<sup>b</sup> Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

<sup>c</sup> Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

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

<sup>e</sup> Interference by co-eluting compounds prevented accurate measurement of analyte.

<sup>f</sup> Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 22

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES  
IN FISH TISSUE (METHOD 5032)<sup>a</sup>

Compound	Efficiency	
	Mean (%)	RSD (%)
Chloromethane	N/A <sup>b</sup>	
Bromomethane	N/A <sup>b</sup>	
Vinyl chloride	N/A <sup>b</sup>	
Chloroethane	N/A <sup>b</sup>	
Methylene chloride	CONT <sup>c</sup>	
Acetone	CONT <sup>c</sup>	
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 <sup>d</sup>	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT <sup>d</sup>	
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 <sup>d</sup>	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT <sup>c</sup>	
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 <sub>8</sub>	88	24
Bromofluorobenzene	52	15

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



TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER  
USING VACUUM DISTILLATION (METHOD 5032)<sup>a</sup>

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

<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 24

VOLATILE ORGANIC ANALYTE RECOVERY FROM FISH OIL  
USING VACUUM DISTILLATION (METHOD 5032)<sup>a</sup>

Compound	Recovery	
	Mean (%)	RSD (%)
Chloromethane	N/A <sup>b</sup>	
Bromomethane	N/A <sup>b</sup>	
Vinyl chloride	N/A <sup>b</sup>	
Chloroethane	N/A <sup>b</sup>	
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 <sup>c</sup>	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT <sup>c</sup>	
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 <sup>c</sup>	
4-Methyl-2-pentanone	INT <sup>c</sup>	
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 <sub>4</sub>	137	30
Toluene-d <sub>8</sub>	84	6
Bromofluorobenzene	48	2

<sup>a</sup> 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.

<sup>b</sup> Not analyzed.

<sup>c</sup> 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)<sup>a</sup>

Compound	Lower Limit of Quantitation (ppb)	
	External Standard Method	Internal Standard Method
Chloromethane	N/A <sup>b</sup>	N/A <sup>b</sup>
Bromomethane	N/A <sup>b</sup>	N/A <sup>b</sup>
Vinyl chloride	N/A <sup>b</sup>	N/A <sup>b</sup>
Chloroethane	N/A <sup>b</sup>	N/A <sup>b</sup>
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 <sup>c</sup>	INT <sup>c</sup>
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT <sup>c</sup>	INT <sup>c</sup>
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 <sup>c</sup>	INT <sup>c</sup>
4-Methyl-2-pentanone	INT <sup>c</sup>	INT <sup>c</sup>
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.)

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- <sup>a</sup> 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.
- <sup>b</sup> No analyses.
- <sup>c</sup> 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 <sub>1</sub>	1,1,2-TCA-d <sub>3</sub>	Bromobenzene-d <sub>5</sub>
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<sup>a</sup> (METHOD 5021)

Compound	% RSD	MQL ( $\mu\text{g}/\text{kg}$ )
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 <sup>b</sup>
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 <sup>c</sup>
Naphthalene	16.8	3.4 <sup>c</sup>
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 <sup>c</sup>
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

- <sup>a</sup> Most compounds spiked at 2 ng/g (2  $\mu\text{g}/\text{kg}$ )  
<sup>b</sup> Incorrect ionization due to methanol  
<sup>c</sup> 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 <sup>a</sup>
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 <sup>a</sup>
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 <sup>a</sup>
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8 <sup>a</sup>
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 <sup>a</sup>
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

<sup>a</sup> 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)<sup>a</sup>

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.)

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- \* 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)

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Bromochloromethane

Acetone  
Acrylonitrile  
Bromomethane  
Carbon disulfide  
Chloroethane  
Chloroform  
Chloromethane  
1,1-Dichloroethane  
1,2-Dichloroethane  
1,2-Dichloroethane-d<sub>4</sub> (surrogate)  
1,1-Dichloroethene  
Trichloroethene  
trans-1,2-Dichloroethene  
Iodomethane  
Methylene chloride  
Trichlorofluoromethane  
Vinyl chloride

Chlorobenzene-d<sub>5</sub>

4-Bromofluorobenzene (surrogate)  
Chlorobenzene  
Ethylbenzene  
Styrene  
1,1,2,2-Tetrachloroethane  
Tetrachloroethene  
Toluene  
Toluene-d<sub>8</sub> (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 <sup>a</sup> (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

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