METHOD 8080

ORGANOCHLORINE PESTICIDES AND PCBs

1.0 SCOPE AND APPLICATION

1.1 Method 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). Table 1 indicates compounds that may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8080 provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides and PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2- to 5-uL sample is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD) or a halogen-specific detector (HSD).

2.2 The sensitivity of Method 8080 usually depends on the level of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8080 may also be performed on samples that have undergone cleanup. Method 3620, Florisil Column Cleanup, by itself or followed by Method 3660, Sulfur Cleanup, may be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Refer to Methods 3500 (Section 3.5, in particular), 3600, and 8000.

3.2 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks, especially in the 15% and 50% fractions from the Florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. The contamination from phthalate esters can be completely eliminated with a microcoulometric or electrolytic conductivity detector.

TABLE 1. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs[a]

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method Detection limit (ug/L)
Aldrin	2.40	4.10	0.004
α-BHC	1.35	1.82	0.003
β-BHC	1.90	1.97	0.006
δ-BHC	2.15	2.20	0.009
γ-BHC (Lindane)	1.70	2.13	0.004
Chlordane (technical)	e	e	0.014
4,4'-DDD	7.83	9.08	0.011
4,4'-DDE	5.13	7.15	0.004
4,4'-DDT	9.40	11.75	0.012
Dieldrin	5.45	7.23	0.002
Endosulfan I	4.50	6.20	0.014
Endosulfan II	8.00	8.28	0.004
Endosulfan sulfate	14.22	10.70	0.066
Endrin	6.55	8.10	0.006
Endrin aldehyde	11.82	9.30	0.023
Heptachlor	2.00	3.35	0.003
Heptachlor epoxide	3.50	5.00	0.083
Methoxychlor	18.20	26.60	0.176
Toxaphene	e	e	0.24
PCB-1016	e	e	nd
PCB-1221	e	e	nd
PCB-1232	e	e	nd
PCB-1242	e	e	0.065
PCB-1248	e	e	nd
PCB-1254	e	e	nd
PCB-1260	e	e	nd

[a]U.S. EPA. Method 617. Organochloride Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

e = Multiple peak response.

nd = not determined.

METHOD 8250

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR SEMIVOLATILE O
PACKED COLUMN TECHNIQUE

T-6288

1.0 SCOPE AND APPLICATION

1.1 Method 8250 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications.

1.2 Method 8250 can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic packed column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Under the alkaline conditions of the extraction step, α-BHC, γ-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected and are not being determined by Method 8080. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4 The practical quantitation limit (PQL) of Method 8250 for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 ug/L for ground water samples (see Table 2). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

METHOD 8250

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR SEMIVOLATILE ORGANICS: PACKED COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Method 8250 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications.

1.2 Method 8250 can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic packed column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

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1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

TABLE 1. CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Method detection limit (ug/L)	Primary Ion	Secondary Ion(s)
Acenaphthene	17.8	1.9	154	153, 152
Acenaphthene-d_{10} (I.S.)	--	--	164	162, 160
Acenaphthylene	17.4	3.5	152	151, 153
Acetophenone	--	--	105	77, 51
Aldrin	24.0	1.9	66	263, 220
Aniline	--	--	93	66, 65
Anthracene	22.8	1.9	178	176, 179
4-Aminobiphenyl	--	--	169	168, 170
Aroclor-1016[b]	18-30	--	222	260, 292
Aroclor-1221[b]	15-30	30	190	224, 260
Aroclor-1232[b]	15-32	--	190	224, 260
Aroclor-1242[b]	15-32	--	222	256, 292
Aroclor-1248[b]	12-34	--	292	362, 326
Aroclor-1254[b]	22-34	36	292	362, 326
Aroclor-1260[b]	23-32	--	360	362, 394
Benzidine[a]	28.8	44	184	92, 185
Benzoic acid	--	--	122	105, 77
Benzo(a)anthracene	31.5	7.8	228	229, 226
Benzo(b)fluoranthene	34.9	4.8	252	253, 125
Benzo(k)fluoranthene	34.9	2.5	252	253, 125
Benzo(g,h,i)perylene	45.1	4.1	276	138, 277
Benzo(a)pyrene	36.4	2.5	252	253, 125
Benzyl alcohol	--	--	108	79, 77
α-BHC[a]	21.1	--	183	181, 109
β-BHC	23.4	4.2	181	183, 109
δ-BHC	23.7	3.1	183	181, 109
γ-BHC (Lindane)[a]	22.4	--	183	181, 109
Bis(2-chloroethoxy)methane	12.2	5.3	93	95, 123
Bis(2-chloroethyl)ether	8.4	5.7	93	63, 95
Bis(2-chloroisopropyl)ether	9.3	5.7	45	77, 121
Bis(2-ethylhexyl)phthalate	30.6	2.5	149	167, 279
4-Bromophenyl phenyl ether	21.2	1.9	248	250, 141
Butyl benzyl phthalate	29.9	2.5	149	91, 206
Chlordane[b]	19-30	--	373	375, 377
4-Chloroaniline	--	--	127	129
1-Chloronaphthalene	--	--	162	127, 164
2-Chloronaphthalene	15.9	1.9	162	127, 164
4-Chloro-3-methylphenol	13.2	3.0	107	144, 142
2-Chlorophenol	5.9	3.3	128	64, 130
4-Chlorophenyl phenyl ether	19.5	4.2	204	206, 141
Chrysene	31.5	2.5	228	226, 229
Chrysene-d_{12} (I.S.)	--	--	240	120, 236
4,4'-DDD	28.6	2.8	235	237, 165
4,4'-DDE	27.2	5.6	246	248, 176

TABLE 1. - Continued

Compound	Retention Time (min)	Method detection limit (ug/L)	Primary Ion	Secondary Ion(s)
4,4'-DDT	29.3	4.7	235	237, 165
Dibenz(a,j)acridine	--	--	279	280, 277
Dibenz(a,h)anthracene	43.2	2.5	278	139, 279
Dibenzofuran	--	--	168	139
Di-n-butylphthalate	24.7	2.5	149	150, 104
1,3-Dichlorobenzene	7.4	1.9	146	148, 111
1,4-Dichlorobenzene	7.8	4.4	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	--	--	152	150, 115
1,2-Dichlorobenzene	8.4	1.9	146	148, 111
3,3'-Dichlorobenzidine	32.2	16.5	252	254, 126
2,4-Dichlorophenol	9.8	2.7	162	164, 98
2,6-Dichlorophenol	--	--	162	164, 98
Dieldrin	27.2	2.5	79	263, 279
Diethylphthalate	20.1	1.9	149	177, 150
p-Dimethylaminoazobenzene	--	--	120	225, 77
7,12-Dimethylbenz(a)anthracene	--	--	256	241, 257
α-,α-Dimethylphenethylamine	--	--	58	91, 42
2,4-Dimethylphenol	9.4	2.7	122	107, 121
Dimethylphthalate	18.3	1.6	163	194, 164
4,6-Dinitro-2-methylphenol	16.2	24	198	51, 105
2,4-Dinitrophenol	15.9	42	184	63, 154
2,4-Dinitrotoluene	19.8	5.7	165	63, 89
2,6-Dinitrotoluene	18.7	1.9	165	63, 89
Diphenylamine	--	--	169	168, 167
1,2-Diphenylhydrazine	--	--	77	105, 182
Di-n-octylphthalate	32.5	2.5	149	167, 43
Endosulfan I[a]	26.4	--	195	339, 341
Endosulfan II[a]	28.6	--	337	339, 341
Endosulfan sulfate	29.8	5.6	272	387, 422
Endrin[a]	27.9	--	263	82, 81
Endrin aldehyde	--	--	67	345, 250
Endrin ketone	--	--	317	67, 319
Ethyl methanesulfonate	--	--	79	109, 97
Fluoranthene	26.5	2.2	202	101, 203
Fluorene	19.5	1.9	166	165, 167
2-Fluorobiphenyl (surr.)	--	--	172	171
2-Fluorophenol (surr.)	--	--	112	64
Heptachlor	23.4	1.9	100	272, 274
Heptachlor epoxide	25.6	2.2	353	355, 351
Hexachlorobenzene	21.0	1.9	284	142, 249
Hexachlorobutadiene	11.4	0.9	225	223, 227
Hexachlorocyclopentadiene[a]	13.9	--	237	235, 272
Hexachloroethane	8.4	1.6	117	201, 199
Indeno(1,2,3-cd)pyrene	42.7	3.7	276	138, 227

TABLE 1. - Continued

Compound	Retention Time (min)	Method detection limit (ug/L)	Primary Ion	Secondary Ion(s)
Isophorone	11.9	2.2	82	95, 138
Methoxychlor	--	--	227	228
3-Methylcholanthrene	--	--	268	253, 267
Methyl methanesulfonate	--	--	80	79, 65
2-Methylnaphthalene	--	--	142	141
2-Methylphenol	--	--	108	107, 79
4-Methylphenol	--	--	108	107, 79
Naphthalene	12.1	1.6	128	129, 127
Naphthalene-d_8 (I.S.)	--	--	136	68
1-Naphthylamine	--	--	143	115, 116
2-Naphthylamine	--	--	143	115, 116
2-Nitroaniline	--	--	65	92, 138
3-Nitroaniline	--	--	138	108, 92
4-Nitroaniline	--	--	138	108, 92
Nitrobenzene	11.1	1.9	77	123, 65
Nitrobenzene-d_5 (surr.)	--	--	82	128, 54
2-Nitrophenol	6.5	3.6	139	109, 65
4-Nitrophenol	20.3	2.4	139	109, 65
N-Nitroso-di-n-butylamine	--	--	84	57, 41
N-Nitrosodimethylamine[a]	--	--	42	74, 44
N-Nitrosodiphenylamine[a]	20.5	1.9	169	168, 167
N-Nitroso-di-N-propylamine	--	--	70	130, 42
N-Nitrosopiperidine	--	--	42	114, 55
Pentachlorobenzene	--	--	250	252, 248
Pentachloronitrobenzene	--	--	295	237, 142
Pentachlorophenol	17.5	3.6	266	264, 268
Perylene-d_{12} (I.S.)	--	--	264	260, 265
Phenacetin	--	--	108	109, 179
Phenanthrene	22.8	5.4	178	179, 176
Phenanthrene-d_{10} (I.S.)	--	--	188	94, 80
Phenol	8.0	1.5	94	65, 66
Phenol-d_6 (surr.)	--	--	99	42, 71
2-Picoline	--	--	93	66, 92
Pronamide	--	--	173	175, 145
Pyrene	27.3	1.9	202	200, 203
Terphenyl-d_{14} (surr.)	--	--	244	122, 212
1,2,4,5-Tetrachlorobenzene	--	--	216	214, 218
2,3,4,6-Tetrachlorophenol	--	--	232	230, 131
2,4,6-Tribromophenol (surr.)	--	--	330	332, 141
1,2,4-Trichlorobenzene	11.6	1.9	180	182, 145
2,4,5-Trichlorophenol	--	--	196	198, 200
2,4,6-Trichlorophenol	11.8	2.7	196	198, 200
Toxaphene[b]	25-34	--	159	231, 233

[a]See Section 1.3
[b]These compounds are mixtures of various isomers.

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system:

4.1.1 **Gas chromatograph:** An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases.

4.1.2 **Columns:**

4.1.2.1 For base/neutral compound detection: 2-m x 2-mm I.D. stainless or glass, packed with 3% SP-2250-DB on 100/120 mesh Supelcoport or equivalent.

4.1.2.2 For acid compound detection: 2-m x 2-mm I.D. glass, packed with 1% SP-1240-DA on 100/120 mesh Supelcoport or equivalent.

4.1.3 **Mass spectrometer:** Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 3 when 1 uL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.4 **GC/MS interface:** Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be

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Acetophenone	--	--	105	77, 51
Aldrin	24.0	1.9	66	263, 220
Aniline	--	--	93	66, 65
Anthracene	22.8	1.9	178	176, 179
4-Aminobiphenyl	--	--	169	168, 170
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Aroclor-1232[b]	15-32	--	190	224, 260
Aroclor-1242[b]	15-32	--	222	256, 292
Aroclor-1248[b]	12-34	--	292	362, 326
Aroclor-1254[b]	22-34	36	292	362, 326
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Benzidine[a]	28.8	44	184	92, 185
Benzoic acid	--	--	122	105, 77
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Benzo(b)fluoranthene	34.9	4.8	252	253, 125
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Benzo(g,h,i)perylene	45.1	4.1	276	138, 277
Benzo(a)pyrene	36.4	2.5	252	253, 125
Benzyl alcohol	--	--	108	79, 77
α-BHC[a]	21.1	--	183	181, 109
β-BHC	23.4	4.2	181	183, 109
δ-BHC	23.7	3.1	183	181, 109
γ-BHC (Lindane)[a]	22.4	--	183	181, 109
Bis(2-chloroethoxy)methane	12.2	5.3	93	95, 123
Bis(2-chloroethyl)ether	8.4	5.7	93	63, 95
Bis(2-chloroisopropyl)ether	9.3	5.7	45	77, 121
Bis(2-ethylhexyl)phthalate	30.6	2.5	149	167, 279
4-Bromophenyl phenyl ether	21.2	1.9	248	250, 141
Butyl benzyl phthalate	29.9	2.5	149	91, 206
Chlordane[b]	19-30	--	373	375, 377
4-Chloroaniline	--	--	127	129
1-Chloronaphthalene	--	--	162	127, 164
2-Chloronaphthalene	15.9	1.9	162	127, 164
4-Chloro-3-methylphenol	13.2	3.0	107	144, 142
2-Chlorophenol	5.9	3.3	128	64, 130
4-Chlorophenyl phenyl ether	19.5	4.2	204	206, 141
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Dibenzofuran	--	--	168	139
Di-n-butylphthalate	24.7	2.5	149	150, 104
1,3-Dichlorobenzene	7.4	1.9	146	148, 111
1,4-Dichlorobenzene	7.8	4.4	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	--	--	152	150, 115
1,2-Dichlorobenzene	8.4	1.9	146	148, 111
3,3'-Dichlorobenzidine	32.2	16.5	252	254, 126
2,4-Dichlorophenol	9.8	2.7	162	164, 98
2,6-Dichlorophenol	--	--	162	164, 98
Dieldrin	27.2	2.5	79	263, 279
Diethylphthalate	20.1	1.9	149	177, 150
p-Dimethylaminoazobenzene	--	--	120	225, 77
7,12-Dimethylbenz(a)anthracene	--	--	256	241, 257
α-,α-Dimethylphenethylamine	--	--	58	91, 42
2,4-Dimethylphenol	9.4	2.7	122	107, 121
Dimethylphthalate	18.3	1.6	163	194, 164
4,6-Dinitro-2-methylphenol	16.2	24	198	51, 105
2,4-Dinitrophenol	15.9	42	184	63, 154
2,4-Dinitrotoluene	19.8	5.7	165	63, 89
2,6-Dinitrotoluene	18.7	1.9	165	63, 89
Diphenylamine	--	--	169	168, 167
1,2-Diphenylhydrazine	--	--	77	105, 182
Di-n-octylphthalate	32.5	2.5	149	167, 43
Endosulfan I[a]	26.4	--	195	339, 341
Endosulfan II[a]	28.6	--	337	339, 341
Endosulfan sulfate	29.8	5.6	272	387, 422
Endrin[a]	27.9	--	263	82, 81
Endrin aldehyde	--	--	67	345, 250
Endrin ketone	--	--	317	67, 319
Ethyl methanesulfonate	--	--	79	109, 97
Fluoranthene	26.5	2.2	202	101, 203
Fluorene	19.5	1.9	166	165, 167
2-Fluorobiphenyl (surr.)	--	--	172	171
2-Fluorophenol (surr.)	--	--	112	64
Heptachlor	23.4	1.9	100	272, 274
Heptachlor epoxide	25.6	2.2	353	355, 351
Hexachlorobenzene	21.0	1.9	284	142, 249
Hexachlorobutadiene	11.4	0.9	225	223, 227
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Indeno(1,2,3-cd)pyrene	42.7	3.7	276	138, 227

TABLE 1. - Continued

Compound	Retention Time (min)	Method detection limit (ug/L)	Primary Ion	Secondary Ion(s)
Isophorone	11.9	2.2	82	95, 138
Methoxychlor	--	--	227	228
3-Methylcholanthrene	--	--	268	253, 267
Methyl methanesulfonate	--	--	80	79, 65
2-Methylnaphthalene	--	--	142	141
2-Methylphenol	--	--	108	107, 79
4-Methylphenol	--	--	108	107, 79
Naphthalene	12.1	1.6	128	129, 127
Naphthalene-d_8 (I.S.)	--	--	136	68
1-Naphthylamine	--	--	143	115, 116
2-Naphthylamine	--	--	143	115, 116
2-Nitroaniline	--	--	65	92, 138
3-Nitroaniline	--	--	138	108, 92
4-Nitroaniline	--	--	138	108, 92
Nitrobenzene	11.1	1.9	77	123, 65
Nitrobenzene-d_5 (surr.)	--	--	82	128, 54
2-Nitrophenol	6.5	3.6	139	109, 65
4-Nitrophenol	20.3	2.4	139	109, 65
N-Nitroso-di-n-butylamine	--	--	84	57, 41
N-Nitrosodimethylamine[a]	--	--	42	74, 44
N-Nitrosodiphenylamine[a]	20.5	1.9	169	168, 167
N-Nitroso-di-N-propylamine	--	--	70	130, 42
N-Nitrosopiperidine	--	--	42	114, 55
Pentachlorobenzene	--	--	250	252, 248
Pentachloronitrobenzene	--	--	295	237, 142
Pentachlorophenol	17.5	3.6	266	264, 268
Perylene-d_{12} (I.S.)	--	--	264	260, 265
Phenacetin	--	--	108	109, 179
Phenanthrene	22.8	5.4	178	179, 176
Phenanthrene-d_{10} (I.S.)	--	--	188	94, 80
Phenol	8.0	1.5	94	65, 66
Phenol-d_6 (surr.)	--	--	99	42, 71
2-Picoline	--	--	93	66, 92
Pronamide	--	--	173	175, 145
Pyrene	27.3	1.9	202	200, 203
Terphenyl-d_{14} (surr.)	--	--	244	122, 212
1,2,4,5-Tetrachlorobenzene	--	--	216	214, 218
2,3,4,6-Tetrachlorophenol	--	--	232	230, 131
2,4,6-Tribromophenol (surr.)	--	--	330	332, 141
1,2,4-Trichlorobenzene	11.6	1.9	180	182, 145
2,4,5-Trichlorophenol	--	--	196	198, 200
2,4,6-Trichlorophenol	11.8	2.7	196	198, 200
Toxaphene[b]	25-34	--	159	231, 233

[a]See Section 1.3
[b]These compounds are mixtures of various isomers.

TABLE 1. RETENTION TIMES AND CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Acetone	--	43	58
Acrolein	--	56	55, 58
Acrylonitrile	--	53	52, 51
Benzene	17.0	78	52, 77
Bromochloromethane (I.S.)	9.3	128	49, 130, 51
Bromodichloromethane	14.3	83	85, 129
4-Bromofluorobenzene (surr.)	28.3	95	174, 176
Bromoform	19.8	173	171, 175, 252
Bromomethane	3.1	94	96, 79
2-Butanone	--	72	57, 43
Carbon disulfide	--	76	78
Carbon tetrachloride	13.7	117	119, 121
Chlorobenzene	24.6	112	114, 77
Chlorobenzene-d_5 (I.S.)	--	117	82, 119
Chlorodibromomethane	--	129	208, 206
Chloroethane	4.6	64	66, 49
2-Chloroethyl vinyl ether	18.6	63	65, 106
Chloroform	11.4	83	85, 47
Chloromethane	2.3	50	52, 49
Dibromomethane	--	93	174, 95
1,4-Dichloro-2-butane	--	75	53, 89
Dichlorodifluoromethane	--	85	87, 50, 101
1,1-Dichloroethane	--	63	65, 83
1,2-Dichloroethane	10.1	62	64, 98
1,2-Dichloroethane-d_4 (surr.)	12.1	65	102
1,1-Dichloroethene	9.0	96	61, 98
trans-1,2-Dichloroethene	10.0	96	61, 98
1,2-Dichloropropane	15.7	63	62, 41
cis-1,3-Dichloropropene	15.9	75	77, 39
trans-1,3-Dichloropropene	17.2	75	77, 39
1,4-Difluorobenzene (I.S.)	19.6	114	63, 88
Ethanol	--	31	45, 27, 46
Ethylbenzene	26.4	106	91
Ethyl methacrylate	--	69	41, 39, 99
2-Hexanone	--	43	58, 57, 100
Iodomethane	--	142	127, 141
Methylene chloride	6.4	84	49, 51, 86
4-Methyl-2-pentanone	--	43	58, 100
Styrene	--	104	78, 103
1,1,2,2-Tetrachloroethane	22.1	83	85, 131, 133
Tetrachloroethene	22.2	164	129, 131, 166
Toluene	23.5	92	91, 65
Toluene-d_8 (surr.)	--	98	70, 100

TABLE 1. - Continued

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
1,1,1-Trichloroethane	13.4	97	99, 117
1,1,2-Trichloroethane	17.2	97	83, 85, 99
Trichloroethene	16.5	130	95, 97, 132
Trichlorofluoromethane	8.3	101	103, 66
1,2,3-Trichloropropane	--	75	110, 77, 61
Vinyl acetate	--	43	86
Vinyl chloride	3.8	62	64, 61
Xylene	--	106	91

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR SEMIVOLATILE ORGANICS: CAPILLARY COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications.

1.2 Method 8270 can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Under the alkaline conditions of the extraction step, α-BHC, γ-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4 The practical quantitation limit (PQL) of Method 8270 for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 ug/L for ground water samples (see Table 2). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

TABLE 1. CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Acenaphthene	15.13	154	153, 152
Acenaphthene-d_{10} (I.S.)	15.05	164	162, 160
Acenaphthylene	14.57	152	151, 153
Acetophenone	7.96[a]	105	77, 51
Aldrin	--	66	263, 220
Aniline	5.68	93	66, 65
Anthracene	19.77	178	176, 179
4-Aminobiphenyl	19.18[a]	169	168, 170
Aroclor-1016	--	222	260, 292
Aroclor-1221	--	190	224, 260
Aroclor-1232	--	190	224, 260
Aroclor-1242	--	222	256, 292
Aroclor-1248	--	292	362, 326
Aroclor-1254	--	292	362, 326
Aroclor-1260	--	360	362, 394
Benzidine	23.87	184	92, 185
Benzoic acid	9.38	122	105, 77
Benzo(a)anthracene	27.83	228	229, 226
Benzo(b)fluoranthene	31.45	252	253, 125
Benzo(k)fluoranthene	31.55	252	253, 125
Benzo(g,h,i)perylene	41.43	276	138, 277
Benzo(a)pyrene	32.80	252	253, 125
Benzyl alcohol	6.78	108	79, 77
α-BHC	--	183	181, 109
β-BHC	--	181	183, 109
δ-BHC	--	183	181, 109
γ-BHC (Lindane)	--	183	181, 109
Bis(2-chloroethoxy)methane	9.23	93	95, 123
Bis(2-chloroethyl)ether	5.82	93	63, 95
Bis(2-chloroisopropyl)ether	7.22	45	77, 121
Bis(2-ethylhexyl)phthalate	28.47	149	167, 279
4-Bromophenyl phenyl ether	18.27	248	250, 141
Butyl benzyl phthalate	26.43	149	91, 206
Chlordane	--	373	375, 377
4-Chloroaniline	10.08	127	129
1-Chloronaphthalene	13.65[a]	162	127, 164
2-Chloronaphthalene	13.30	162	127, 164
4-Chloro-3-methylphenol	11.68	107	144, 142
2-Chlorophenol	5.97	128	64, 130
4-Chlorophenyl phenyl ether	16.78	204	206, 141
Chrysene	27.97	228	226, 229
Chrysene-d_{12} (I.S.)	27.88	240	120, 236
4,4'-DDD	--	235	237, 165
4,4'-DDE	--	246	248, 176

TABLE 1. CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
4,4'-DDT	--	235	237, 165
Dibenz(a,j)acridine	32.55[a]	279	280, 277
Dibenz(a,h)anthracene	39.82	278	139, 279
Dibenzofuran	15.63	168	139
Di-n-butylphthalate	21.78	149	150, 104
1,3-Dichlorobenzene	6.27	146	148, 111
1,4-Dichlorobenzene	6.40	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	6.35	152	150, 115
1,2-Dichlorobenzene	6.85	146	148, 111
3,3'-Dichlorobenzidine	27.88	252	254, 126
2,4-Dichlorophenol	9.48	162	164, 98
2,6-Dichlorophenol	10.05[a]	162	164, 98
Dieldrin	--	79	263, 279
Diethylphthalate	16.70	149	177, 150
p-Dimethylaminoazobenzene	24.48[a]	120	225, 77
7,12-Dimethylbenz(a)anthracene	29.54[a]	256	241, 257
α-,α-Dimethylphenethylamine	9.51[a]	58	91, 42
2,4-Dimethylphenol	9.03	122	107, 121
Dimethylphthalate	14.48	163	194, 164
4,6-Dinitro-2-methylphenol	17.05	198	51, 105
2,4-Dinitrophenol	15.35	184	63, 154
2,4-Dinitrotoluene	15.80	165	63, 89
2,6-Dinitrotoluene	14.62	165	63, 89
Diphenylamine	17.54[a]	169	168, 167
1,2-Diphenylhydrazine	--	77	105, 182
Di-n-octylphthalate	30.48	149	167, 43
Endosulfan I	--	195	339, 341
Endosulfan II	--	337	339, 341
Endosulfan sulfate	--	272	387, 422
Endrin	--	263	82, 81
Endrin aldehyde	--	67	345, 250
Endrin ketone	--	317	67, 319
Ethyl methanesulfonate	5.33[a]	79	109, 97
Fluoranthene	23.33	202	101, 203
Fluorene	16.70	166	165, 167
2-Fluorobiphenyl (surr.)	--	172	171
2-Fluorophenol (surr.)	--	112	64
Heptachlor	--	100	272, 274
Heptachlor epoxide	--	353	355, 351
Hexachlorobenzene	18.65	284	142, 249
Hexachlorobutadiene	10.43	225	223, 227
Hexachlorocyclopentadiene	12.60	237	235, 272
Hexachloroethane	7.65	117	201, 199
Indeno(1,2,3-cd)pyrene	39.52	276	138, 227

TABLE 1. CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Continued)

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Isophorone	8.53	82	95, 138
Methoxychlor	--	227	228
3-Methylcholanthrene	31.14[a]	268	253, 267
Methyl methanesulfonate	4.32[a]	80	79, 65
2-Methylnaphthalene	11.87	142	141
2-Methylphenol (o-cresol)	7.22	108	107, 79
4-Methylphenol (p-cresol)	7.60	108	107, 79
Naphthalene	9.82	128	129, 127
Naphthalene-d_8 (I.S.)	9.75	136	68
1-Naphthylamine	15.80[a]	143	115, 116
2-Naphthylamine	16.00[a]	143	115, 116
2-Nitroaniline	13.75	65	92, 138
3-Nitroaniline	15.02	138	108, 92
4-Nitroaniline	16.90	138	108, 92
Nitrobenzene	7.87	77	123, 65
Nitrobenzene-d_5 (surr.)	--	82	128, 54
2-Nitrophenol	8.75	139	109, 65
4-Nitrophenol	15.80	139	109, 65
N-Nitroso-di-n-butylamine	10.99[a]	84	57, 41
N-Nitrosodimethylamine	--	42	74, 44
N-Nitrosodiphenylamine	17.17	169	168, 167
N-Nitrosodipropylamine	7.55	70	42, 101, 130
N-Nitrosopiperidine	--	42	114, 55
Pentachlorobenzene	15.64[a]	250	252, 248
Pentachloronitrobenzene	19.47[a]	295	237, 142
Pentachlorophenol	19.25	266	264, 268
Perylene-d_{12} (I.S.)	33.05	264	260, 265
Phenacetin	18.59[a]	108	109, 179
Phenanthrene	19.62	178	179, 176
Phenanthrene-d_{10} (I.S.)	19.55	188	94, 80
Phenol	5.77	94	65, 66
Phenol-d_6 (surr.)	--	99	42, 71
2-Picoline	3.75[a]	93	66, 92
Pronamide	19.61[a]	173	175, 145
Pyrene	24.02	202	200, 203
Terphenyl-d_{14} (surr.)	--	244	122, 212
1,2,4,5-Tetrachlorobenzene	13.62[a]	216	214, 218
2,3,4,6-Tetrachlorophenol	16.09[a]	232	230, 131
2,4,6-Tribromophenol (surr.)	--	330	332, 141
1,2,4-Trichlorobenzene	9.67	180	182, 145
2,4,5-Trichlorophenol	13.00	196	198, 200
2,4,6-Trichlorophenol	12.85	196	198, 200
Toxaphene	--	159	231, 233

I.S. = internal standard
surr. = surrogate

[a]Estimated retention times.

TABLE 2. PRACTICAL QUANTITATION LIMITS (PQL) FOR SEMIVOLATILE ORGANICS**

Semivolatiles	CAS Number	Practical Quantitation Limits*	
		Ground Water	Low Soil/Sediment[1]
		ug/L	ug/Kg
Phenol	108-95-2	10	660
bis(2-Chloroethyl) ether	111-44-4	10	660
2-Chlorophenol	95-57-8	10	660
1,3-Dichlorobenzene	541-73-1	10	660
1,4-Dichlorobenzene	106-46-7	10	660
Benzyl Alcohol	100-51-6	20	1300
1,2-Dichlorobenzene	95-50-1	10	660
2-Methylphenol	95-48-7	10	660
bis(2-Chloroisopropyl) ether	39638-32-9	10	660
4-Methylphenol	106-44-5	10	660
N-Nitroso-Di-N-propylamine	621-64-7	10	660
Hexachloroethane	67-72-1	10	660
Nitrobenzene	98-95-3	10	660
Isophorone	78-59-1	10	660
2-Nitrophenol	88-75-5	10	660
2,4-Dimethylphenol	105-67-9	10	660
Benzoic Acid	65-85-0	50	3300
bis(2-Chloroethoxy) methane	111-91-1	10	660
2,4-Dichlorophenol	120-83-2	10	660
1,2,4-Trichlorobenzene	120-82-1	10	660
Naphthalene	91-20-3	10	660
4-Chloroaniline	106-47-8	20	1300
Hexachlorobutadiene	87-68-3	10	660
4-Chloro-3-methylphenol	59-50-7	20	1300
2-Methylnaphthalene	91-57-6	10	660
Hexachlorocyclopentadiene	77-47-4	10	660
2,4,6-Trichlorophenol	88-06-2	10	660
2,4,5-Trichlorophenol	95-95-4	10	660

TABLE 2. PRACTICAL QUANTITATION LIMITS (PQL) FOR SEMIVOLATILE ORGANICS** (Continued)

Semivolatiles	CAS Number	Practical Quantitation Limits*	
		Ground Water	Low Soil/Sediment[1]
		ug/L	ug/Kg
2-Chloronaphthalene	91-58-7	10	660
2-Nitroaniline	88-74-4	50	3300
Dimethyl phthalate	131-11-3	10	660
Acenaphthylene	208-96-8	10	660
3-Nitroaniline	99-09-2	50	3300
Acenaphthene	83-32-9	10	660
2,4-Dinitrophenol	51-28-5	50	3300
4-Nitrophenol	100-02-7	50	3300
Dibenzofuran	132-64-9	10	660
2,4-Dinitrotoluene	121-14-2	10	660
2,6-Dinitrotoluene	606-20-2	10	660
Diethylphthalate	84-66-2	10	660
4-Chlorophenyl phenyl ether	7005-72-3	10	660
Fluorene	86-73-7	10	660
4-Nitroaniline	100-01-6	50	3300
4,6-Dinitro-2-methylphenol	534-52-1	50	3300
N-Nitrosodiphenylamine	86-30-6	10	660
4-Bromophenyl phenyl ether	101-55-3	10	660
Hexachlorobenzene	118-74-1	10	660
Pentachlorophenol	87-86-5	50	3300
Phenanthrene	85-01-8	10	660
Anthracene	120-12-7	10	660
Di-n-butylphthalate	84-74-2	10	660
Fluoranthene	206-44-0	10	660
Pyrene	129-00-0	10	660
Butyl benzyl phthalate	85-68-7	10	660
3,3'-Dichlorobenzidine	91-94-1	20	1300
Benzo(a)anthracene	56-55-3	10	660
bis(2-ethylhexyl)phthalate	117-81-7	10	660

TABLE 2. PRACTICAL QUANTITATION LIMITS (PQL) FOR SEMIVOLATILE ORGANICS** (Continued)

Semi-Volatiles	CAS Number	Practical Quantitation Limits*	
		Ground Water	Low Soil/Sediment[1]
		ug/L	ug/Kg
Chrysene	218-01-9	10	660
Di-n-octyl phthalate	117-84-0	10	660
Benzo(b)fluoranthene	205-99-2	10	660
Benzo(k)fluoranthene	207-08-9	10	660
Benzo(a)pyrene	50-32-8	10	660
Indeno(1,2,3-cd)pyrene	193-39-5	10	660
Dibenz(a,h)anthracene	53-70-3	10	660
Benzo(g,h,i)perylene	191-24-2	10	660

*PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis, therefore, PQLs will be higher based on the % moisture in each sample. This is based on a 30-g sample and gel permeation chromatography cleanup.

**Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achieveable.

Other Matrices	Factor[1]
Medium-level soil and sludges by sonicator	7.5
Non-water-miscible waste	75

[1]PQL = [PQL for Ground Water (Table 2)] X [Factor].

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system:

4.1.1 **Gas chromatograph:** An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 **Column:** 30-m x 0.25-mm I.D. (or 0.32-mm I.D.) 1-um film thickness silicon-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 **Mass spectrometer:** Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 3 when 1 uL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.4 **GC/MS interface:** Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

4.1.5 **Data system:** A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have