VOLATILE ORGANIC COMPOUNDS (VOCs)

SW-846 Method 8260

Table 1A. Summary of Holding Times and Preservation for Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Analytical Parameter [a]	Technical and Contract Holding Times	Preservation
Volatile Organic Compounds (VOCs) in Water	Technical: 7 days from collection; Contract: 5 days from receipt at laboratory	Cool to 4EC ±2EC;
VOCs in Water	Technical: 14 days from collection; Contract: 10 days from receipt at laboratory	HCl to pH <2; Cool to 4EC ±2EC
VOCs in Soil	Technical: 48 hours Contract: 48 hours	Cool to 4EC ±2EC; sealed zero headspace containers; freezing can extend the holding time [b]
VOCs in Soil	Technical: 14 days from collection; Contract: 10 days from receipt at laboratory	Preserved samples: in methanol [c] or sodium bisulfate [d]

[a] Individual target compounds are listed in Table 1B.
[b] Freezing the sample can extend the holding time; however, 48 hours unfrozen holding time will be considered cumulative.
[c] Use Method 5030 for purge and trap.
[d] Use Method 5035 for purge and trap.

Data Calculations and Reporting Units:

Calculate the response factor (RF) and the concentration of individual analytes according to the equations specified in Sections 7.3.4 of Method 8260. Report water sample results in concentration units of micrograms per liter (Fg/L).

Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits for Volatile Organic Compounds by Method 8260

Analyte	CAS Number	CRQL µg/L[a]	CRQL µg/Kg[b]
Benzene	71-43-2	1	5
Bromobenzene	108-86-1	1	5
Bromochloromethane	74-97-5	1	5
Bromodichloromethane	75-27-4	1	5
Bromoform	75-25-2	1	5
Bromomethane	74-83-9	1	5
n-Butylbenzene	104-51-8	1	5
sec-Butylbenzene	135-98-8	1	5
tert-Butylbenzene	98-06-6	1	5
Carbon tetrachloride	56-23-5	1	5
Chlorobenzene	108-90-7	1	5
Chlorodibromomethane	124-48-1	1	5
Chloroethane	75-00-3	1	5
Chloroform	67-66-3	1	5
Chloromethane	74-87-3	1	5
2-Chlorotoluene	95-49-8	1	5
4-Chlorotoluene	106-43-4	1	5
1,2-Dibromo-3-chloropropane	96-12-8	1	5
1,2-Dibromoethane	106-93-4	1	5
Dibromomethane	74-95-3	1	5
1,2-Dichlorobenzene	95-50-1	1	5
1,3-Dichlorobenzene	541-73-1	1	5
1,4-Dichlorobenzene	106-46-7	1	5
Dichlorodifluoromethane	75-71-8	1	5
1,1-Dichloroethane	75-34-3	1	5
1,2-Dichloroethane	107-06-2	1	5
1,1-Dichloroethene	75-35-4	1	5
cis-1,2-Dichloroethene	156-59-2	1	5
trans-1,2-Dichloroethene	156-60-5	1	5

1,2-Dichloropropane	78-87-5	1	5
2,2-Dichloropropane	594-20-7	1	5
1,3-Dichloropropane	142-28-9	1	5
1,1-Dichloropropene	563-58-6	1	5
Ethylbenzene	100-41-4	1	5
Hexachlorobutadiene	87-68-3	1	5
Isopropylbenzene	98-82-8	1	5
p-Isopropyltoluene	99-87-8	1	5
Methylene chloride	75-09-2	1	5
Naphthalene	91-20-3	1	5
n-Propylbenzene	103-65-1	1	5
Styrene	100-42-5	1	5
1,1,1,2-Tetrachloroethane	630-20-6	1	5
1,1,2,2-Tetrachloroethane	79-34-5	1	5
Tetrachloroethene	127-18-4	1	5
Toluene	108-88-3	1	5
1,2,4-Trichlorobenzene	120-82-1	1	5
1,2,3-Trichlorobenzene	87-61-6	1	5
1,1,1-Trichloroethane	71-55-6	1	5
1,1,2-Trichloroethane	79-00-5	1	5
Trichloroethene	79-01-6	1	5
Trichlorofluoromethane	75-69-4	1	5
1,2,3-Trichloropropane	96-18-4	1	5
1,2,4-Trimethylbenzene	95-63-6	1	5
1,3,5-Trimethylbenzene	108-67-8	1	5
Vinyl chloride	75-01-4	1	5
o-Xylene	95-47-6	1	5
m-Xylene	108-38-3	1	5
p-Xylene	106-42-3	1	5
Methyl-t-butyl ether	163-40-44	1	5
Dichlorofluoromethane	75-43-4	1	5

[a] Based on 25 mL water purge. [b] Based on wet weight

Table 2. Summary of Calibration Procedures for VOCs by SW-846 Method 8260

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with BFB	Beginning of each 12 hour period during which standards samples are analyzed	Ion abundance criteria in Table 4 of Method 8260	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL)[a, b, c]	Initially; whenever required, due to failure of CCV	RSD for RFs #20%;	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV)	Following ICV, every 12-hour, and end of run	%D between RF of CCV and avg RFs from ICAL #15%	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
System Performance Check Compound (SPCC)	With ICAL or CCV	RF for chloromethane, 1,1-dichloroethane, bromoform, $0.10; chlorobenzene, 1,1,2,2-tetrachloroethane, $0.30	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Calibration Check Compounds (CCC)	With ICAL or CCV	RSD for RFs #30%	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Internal Standards	Each analysis of CCV	-50 to +100%	1. Re-analyze all samples analyzed while system was out-of-control
Retention time evaluation of CCV standards	Each analysis of CCV standard	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.
[b] ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.
[c] Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation of the average initial calibration retention time for each analyte.

Table 3. Summary of Internal Quality Control Procedures for VOCs by SW-846 Method 8260

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG [a]	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Reanalyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	Water Sample: 65-135% of expected value; #30% RPD between MS and MSD Soil Sample: 50-150% of expected value; #50% RPD between MS and MSD	1. Report in case narrative
Surrogate Spikes:[b]	Every sample, standard and method blank	Water Sample: 85-115% except for 1,2-dichloroethane (75-115%) of expected value Soil Sample: 70-125% of expected value	1. Reanalyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	Water Sample: 70-130% of expected value Soil Sample: 65-135% of expected value	1. Investigate the source of problem and document. 2. Reanalyze all samples processed with a LCS that is out of control.

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.
[b] Toluene-d_8, BFB, 1,2-dichloroethane-d_4, and Dibromofluoromethane

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve.

Report results and submit documentation for both analyses.

METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/
MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

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

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d_5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc

(continued)

TABLE 1

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

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2'[c]	(µg/L)
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)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2"[c]	(µg/L)
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) Column 1[a]	Column 2[b]	Column 2"[c]	MDL[d] (µg/L)
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d_5	23.10			
1,4-Dichlorobenzene-d_4	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d_4	32.30	19.08	27.25	
Dichloroethane-d_4	12.08			
Dibromofluoromethane	--			
Toluene-d_8	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

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

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

[d] MDL based on a 25-mL sample volume.

TABLE 2

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

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

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

[b] MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment[b] µg/kg
5	1	5

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

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

Other Matrices	Factor[c]
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

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

For non-aqueous samples, the factor is on a wet-weight basis.

SEMIVOLATILE ORGANIC COMPOUNDS (SVOCs)

SW-846 Method 8270

Table 1A. Summary of Holding Times and Preservation for Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Analytical Parameter [a]	Technical and Contract Holding Times	Preservation
Semivolatile Organic Compounds (SVOCs) in Water	Technical for Extraction: 14 days from collection; Contract for Extraction: 10 days from receipt at laboratory; Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC;
SVOCs in Soil [b]	Technical for Extraction: 14 days from collection; Contract for Extraction: 10 days from receipt at laboratory; Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC

[a] Individual target compounds are listed in Table 1B.
[b] Perform initial sample analysis using a 2-gram sample for mid-level analysis and a 30-gram sample for low-level analysis

Data Calculations and Reporting Units:

Use the mean RRF from the initial calibration to calculate the concentration of individual analytes according to Section 7.7.2 of EPA Method 8270C, Revision 3.0.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B: Target Compound List and Contract Required Quantitation Limits (CRQLs) for Semivolatile Organic Compounds (SVOCs) by SW-846 Method 8270

Analyte	Contract Required Quantitation Limits		PRGs	
	Water (Fg/L)	Soil (mg/kg)	Industrial Soil (mg/kg)	Tap Water (Fg/L)
1,2-Dichlorobenzene	10	0.33	370	370
1,2,4-Trichlorobenzene	10	0.33	1,700	190
1,3-Dichlorobenzene	10	0.33	140	180
1,4-Dichlorobenzene	10	0.33	7.3	0.47
2-Chloronaphthalene	10	0.33	NA	NA
2-Chlorophenol	10	0.33	240	38
2-Methylnaphthalene	10	0.33	NA	NA
2-Methylphenol	10	0.33	53,000	1,800
2-Nitroaniline	25	0.8	64	2.2
2-Nitrophenol	10	0.33	NA	NA
2,2'-oxybis (1-Chloropropane)	10	0.33	NA	NA
2,4-Dichlorophenol	10	0.33	3,200	110
2,4-Dimethylphenol	10	0.33	21,000	730
2,4-Dinitrophenol	25	0.8	2,100	73
2,4-Dinitrotoluene	10	0.33	2,100	73
2,4,5-Trichlorophenol	25	0.8	110,000	3,700
2,4,6-Trichlorophenol	10	0.33	270	6.1
2,6-Dinitrotoluene	10	0.33	1,100	37
3-Nitroaniline	25	0.8	NA	NA
3,3'-Dichlorobenzidine	10	0.33	6.7	0.15
4-Bromophenyl-phenylether	10	0.33	NA	NA
4-Chloro-3-methylphenol	10	0.33	NA	NA
4-Chloroaniline	10	0.33	4,300	150
4-Chlorophenyl-phenyl ether	10	0.33	NA	NA
4-Methylphenol	10	0.33	5,300	180
4-Nitroaniline	25	0.8	NA	NA
4-Nitrophenol	25	0.8	66,000	2,300
4,6-Dinitro-2-methylphenol	25	0.8	NA	NA
Acenaphthene	10	0.33	28,000	370
Acenaphthylene	10	0.33	NA	NA
Anthracene	10	0.33	220,000	1,800
Benzo(a)anthracene	10	0.33	3.6	0.092
Benzo(a)pyrene	10	0.33	0.36	0.0092
Benzo(b)fluoranthene	10	0.33	3.6	0.092
Benzo(g,h,i)perylene	10	0.33	NA	NA
Benzo(k)fluoranthene	10	0.33	36	0.92
bis(2-Chloroethoxy)-methane	10	0.33	NA	NA

Table 1B: Target Compound List and Contract Required Quantitation Limits (CRQLs) for Semivolatile Organic Compounds (SVOCs) by SW-846 Method 8270

Analyte	Contract Required Quantitation Limits		PRGs	
	Water (Fg/L)	Soil (mg/kg)	Industrial Soil (mg/kg)	Tap Water (Fg/L)
bis(2-Chloroethyl) ether	10	0.33	0.56	0.0098
bis(2-Ethylhexyl)phthalate	10	0.33	21	4.8
Butylbenzylphthalate	10	0.33	930	7,300
Carbazole	10	0.33	150	3.4
Chrysene	10	0.33	360	9.2
Di-n-butylphthalate	10	0.33	NA	NA
Di-n-octylphthalate	10	0.33	10,000	730
Dibenz(a,h)anthracene	10	0.33	0.36	0.0092
Dibenzofuran	10	0.33	3,200	24
Diethylphthalate	10	0.33	100,000	29,000
Dimethylphthalate	10	0.33	100,000	370,000
Fluoranthene	10	0.33	37,000	1,500
Fluorene	10	0.33	22,000	240
Hexachlorobenzene	10	0.33	1.9	0.042
Hexachlorobutadiene	10	0.33	38	0.86
Hexachlorocyclopentadiene	10	0.33	7,100	260
Hexachloroethane	10	0.33	210	4.8
Indeno(1,2,3-cd)pyrene	10	0.33	3.6	0.092
Isophorone	10	0.33	3,200	71
N-Nitroso-di-n-propylamine	10	0.33	0.43	0.0096
N-nitrosodiphenylamine	10	0.33	610	14
Naphthalene	10	0.33	190	6.2
Nitrobenzene	10	0.33	100	3.4
Pentachlorophenol	25	0.8	15	0.56
Phenanthrene	10	0.33	NA	NA
Phenol	10	0.33	100,000	22,000
Pyrene	10	0.33	26,000	180

Notes:

Fg/L = micrograms per liter.
mg/kg = milligrams per kilogram.
NA = Not available.
PRG = U.S. EPA Preliminary Remediation Goals, Region 9 May 1, 1998..

Table 2. Summary of Calibration Procedures for SVOCs by SW-846 Method 8270C

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with decafluorotriphenyl-phosphine (DFTPP)	Beginning of each 12-hour period during which standards samples are analyzed	Ion abundance criteria in Table 3 of Method 8270C, Revision 3.0	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL)[a, b, c]	Initially; whenever required, due to failure of CCV	RSD for RRFs #30%; or correlation coefficient (r) generated by the linear regression must be 0.99 for all analytes	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV) [d]	Beginning of every 12-hour period, and end of run	%D between RRF of CCV and avg RRFs from ICAL #30%; or ±30% of true value for linear regressiion	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Integrated areas of Internal Standards	Each analysis	Area must be within -50 to 100 percent.	1. Re-analyze samples with internal standard -50 percent and greater than 100 percent
Retention time evaluation of all standard, surrogate, and sample analytes	Each analysis	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples out control limits

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.
[b] ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.
[c] Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation of the average initial calibration retention time for each analyte.
[d] If some compounds are beyond the control limits of the CCV and these target compounds are detected in samples and 10 percent or less of these analytes are beyond control limits, a single point calibration may be used to quantify the out-of-control analytes.

Table 3. Summary of Internal Quality Control Procedures for SVOCs by SW-846 Method 8270C

QC Element	Frequency	Acceptance Criteria [b]	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG [a]	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Re-analyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	Water Sample: MS and MSD recoveries and RPD between within laboratory limits Soil Sample: MS and MSD recoveries and RPD between within laboratory limits	1. Report in case narrative
Surrogate Spikes	Every sample, standard and method blank	Water Sample: Surrogate recoveries within laboratory limits Soil Sample: Surrogate recoveries within laboratory limits	1. Re-analyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	Water Sample: LCS recoveries within laboratory limits Soil Sample: LCS recoveries within laboratory limits	1. Investigate the source of problem and document. 2. Re-analyze all samples processed with a LCS that is out of control.

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.
[b] within 3 sigma of laboratory control charts. The laboratory should submit the control charts.

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

METHOD 8270D

SEMIVOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

		Appropriate Preparation Techniques[b]				
Compounds	CAS No[a]	3510	3520	3540/ 3541	3550	3580
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea	591-08-2	LR	ND	ND	ND	LR
Aldrin	309-00-2	X	X	X	X	X
2-Aminoanthraquinone	117-79-3	X	ND	ND	ND	X
Aminoazobenzene	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcarbazole	132-32-1	X	X	ND	ND	ND
Anilazine	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite	140-57-8	HS(43)	ND	ND	ND	X
Aroclor 1016	12674-11-2	X	X	X	X	X
Aroclor 1221	11104-28-2	X	X	X	X	X
Aroclor 1232	11141-16-5	X	X	X	X	X
Aroclor 1242	53469-21-9	X	X	X	X	X
Aroclor 1248	12672-29-6	X	X	X	X	X
Aroclor 1254	11097-69-1	X	X	X	X	X
Aroclor 1260	11096-82-5	X	X	X	X	X
Azinphos-methyl	86-50-0	HS(62)	ND	ND	ND	X
Barban	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP
Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X

describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid device for the extraction of PAHs from solids (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate 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.

In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 may be appropriate for confirmation of the identification of these analytes when concentration in the extract permits. Refer to Sec. 7.0 of Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

1.4 The following compounds may require special treatment when being determined by this method:

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.4.6 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 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.

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. However, the analyst must use caution in modifying the injection port temperature, as the performance of other analytes may be adversely affected. Therefore, if pyridine is to be determined in addition to other target analytes, it may be necessary to perform separate

analyses. In addition, pyridine may be lost during the evaporative concentration of the sample extract. As a result, many of the extraction methods listed above may yield low recoveries unless great care is exercised during the concentration steps. For this reason, analysts may wish to consider the use of extraction techniques such as pressurized fluid extraction (Method 3545) or supercritical fluid extraction, which involve smaller extract volumes, thereby reducing or eliminating the need for evaporative concentration techniques for many applications.

1.4.8 Toluene diisocyanate rapidly hydrolyses in water (half-life of less then 30 min.). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.9 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 2). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 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.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

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

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-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. 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 ID (or 0.32-mm ID) 1-µm film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 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 the criteria in Table 3 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system should 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 should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

Compound	Estimated Quantitation Limits[a]	
	Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
Acenaphthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy)methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-Bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND

TABLE 2
(Continued)

Compound	Estimated Quantitation Limits[a] Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl)pyridine hydrochloride	100	ND
2-Chloronaphthalene	10	660
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-O	10	ND
Demeton-S	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butyl phthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethyl phthalate	10	660
Diethylstilbestrol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
2,4-Dimethylphenol	10	660

TABLE 2
(Continued)

Compound	Estimated Quantitation Limits[a] Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl) phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethylphosphoramide	20	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND

TABLE 2
(Continued)

Compound	Estimated Quantitation Limits[a] Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
Methyl methanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND
4-Methylphenol	10	660
Mevinphos	10	ND
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodi-n-butylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND

TABLE 2
(Continued)

Compound	Estimated Quantitation Limits[a] Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
Octamethyl pyrophosphoramide	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND
Phthalic anhydride	100	ND
2-Picoline	ND	ND
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND

TABLE 2
(Continued)

Compound	Estimated Quantitation Limits[a] Ground water (µg/L)	Low Soil/Sediment[b] (µg/kg)
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND

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

[b] EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined

NA = Not Applicable

Other Matrices	Factor[c]
High-concentration soil and sludges by ultrasonic extractor	7.5
Non-water miscible waste	75

[c]EQL = (EQL for Low Soil/Sediment given above in Table 2) x (Factor)

METHOD 9081

CATION-EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)

1.0 SCOPE AND APPLICATION

1.1 Method 9081 is applicable to most soils, including calcareous and noncalcareous soils. The method of cation-exchange capacity by summation (Chapman, 1965, p. 900; see Paragraph 10.1) should be employed for distinctly acid soils.

2.0 SUMMARY OF METHOD

2.1 The soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by atomic absorption, emission spectroscopy, or an equivalent means.

3.0 INTERFERENCES

3.1 Interferences can occur during analysis of the extract for sodium content. Thoroughly investigate the chosen analytical method for potential interferences.

4.0 APPARATUS AND MATERIALS

4.1 Centrifuge tube and stopper: 50-mL, round-bottom, narrow neck.

4.2 Mechanical shaker.

4.3 Volumetric flask: 100-mL.

5.0 REAGENTS

5.1 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ in water and dilute it to 1,000 mL. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the reaction of the solution to pH 8.2.

5.2 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with water to a volume of approximately 1 liter. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH, as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

5.3 Isopropyl alcohol: 99%.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

7.0 PROCEDURE

7.1 Weigh 4 g of medium- or fine-textured soil or 6 g of coarse-textured soil and transfer the sample to a 50-mL, round-bottom, narrow-neck centrifuge tube. (A fine soil has >50% of the particles ≤0.074 mm, medium soil has >50% ≥0.425 mm, while a coarse soil has more than 50% of its particles ≥2 mm.

7.2 Add 33 mL of 1.0 N NaOAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

7.3 Decant the liquid, and repeat Paragraph 7.2 three more times.

7.4 Add 33 mL of 99% isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

7.5 Repeat the procedure described in Paragraph 7.4 two more times.

7.6 Add 33 mL of NH_4OAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing into a 100-mL volumetric flask.

7.7 Repeat the procedure described in Paragraph 7.6 two more times.

7.8 Dilute the combined washing to the 100-mL mark with ammonium acetate solution and determine the sodium concentration by atomic absorption, emission spectroscopy, or an equivalent method.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

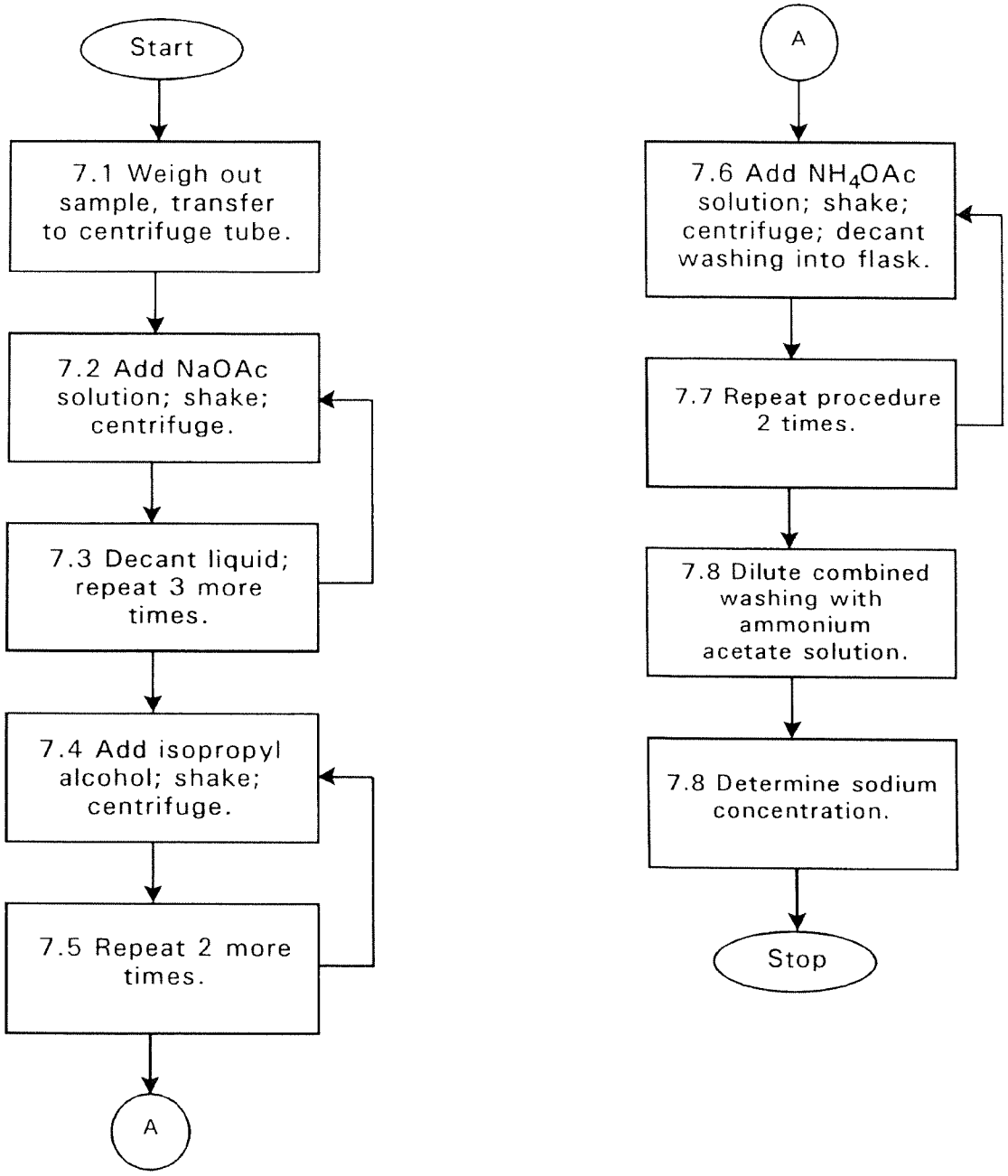
8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Materials of known cation-exchange capacity must be routinely analyzed.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

10.1 This method is based on Chapman, H.D., "Cation-exchange Capacity," pp. 891-900, in C.A. Black (ed.), Method of Soil Analysis, Part 2: Chemical and Microbiological Properties, Am. Soc. Agron., Madison, Wisconsin (1965).

METHOD 9081
CATION-EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)