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Designation: D 6010 – 96

Standard Practice for Closed Vessel Microwave Solvent Extraction of Organic Compounds from Solid Matrices[1]

This standard is issued under the fixed designation D 6010; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the closed vessel microwave extraction of soils, sediments, sludges, and wastes for subsequent determination of solvent extractable semivolatile and nonvolatile organic compounds by such techniques as gas chromatography and gas chromatography-mass spectrometry.

1.1.1 Compounds listed in Tables 1–5 can be extracted from the preceding materials.

1.2 This test method is applicable to samples that will pass through a 10-mesh (approximately 2-mm opening) screen.

1.3 The detection limit and linear concentration range for each compound is dependent on the gas chromatograph or gas chromatograph-mass spectrometer technique employed and may be found in the manual accompanying the instrument used.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 8 for specific hazard statements.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis[3]

D 5368 Test Method for the Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples[4]

2.2 *Other Standards:*

United States Environmental Protection Agency (USEPA), Test Methods for Evaluating Solid Waste Volume 1A: Laboratory Manual Physical/Chemical Methods[5]

Title 21, Code of Federal Regulations (CFR), Part 1030, and Title 47, Part 18[5]

3. Summary of Practice

3.1 This procedure ensures intimate contact of the sample matrix with 115°C extraction solvent.

3.2 A 1 to 5-g portion of a solid sample is extracted in a sealed microwave transparent extraction vessel with 30 mL of acetone-hexane (1 + 1).

3.3 Up to 12 samples may be extracted simultaneously.

3.4 After extraction the vessels are cooled to room temperature, opened, and the solvent and sample are separated by decanting, filtration, or centrifuging.

3.5 This practice provides a sample suitable for analysis by gas chromatography or gas chromatography-mass spectrometry.

4. Significance and Use

4.1 Extraction of organic pollutants from wastes can provide information on the susceptibility of compounds to leeching, water quality changes, or other site conditions.

4.2 Rapid heating, in combination with temperatures in excess of the atmospheric boiling point of organic solvents, reduces sample extraction times.

4.3 Small amounts of solvents (30 mL) are used resulting in reduced sample preparation cost and time.

5. Interferences

5.1 Method interferences may be caused by contaminants in solvents, labware, and other hardware used in sample processing that lead to discrete artifacts or elevated baselines in gas chromatograms. The analyst must demonstrate, through the analysis of reagent blanks, that the system and the materials are free from interferents.

5.2 The use of high-purity solvents helps to minimize interference problems.

5.3 Matrix interferences are caused by contaminants that are coextracted from the sample. The extent of matrix interferences may vary considerably from sample to sample.

5.4 After cleaning, vessel liners and covers should be stored in a clean environment to prevent accumulation of contaminants.

[1] This practice is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.02 on Physical and Chemical Characterization.

Current edition approved Oct. 10, 1996. Published December 1996.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] *Annual Book of ASTM Standards*, Vol 11.04.

[5] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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TABLE 1 Semivolatile Analyte Recovery from Freshly Spiked Topsoil

Analyte	Spike Level, mg/kg	Average Recovery, %[A]	RSD, %
Acenaphthene	5.0	97.6	9.8
Acenaphthylene	5.0	100	10
Acetophenone	5.0	92.2	12
4-Aminobiphenyl	5.0	77.3	9.5
Aniline	5.0	68.1	7.5
Anthracene	5.0	108	9.2
Benzidine	5.0	0	
Benzoic acid	5.0	42.3	13
Benzo(a)anthracene	5.0	113	9.4
Benzo(b)fluoranthene	5.0		
Benzo(k)fluoranthene	5.0	116	9.3
Benzo(g,h,i)perylene	5.0	111	4.7
Benzo(a)pyrene	5.0	110	8.6
Benzyl alcohol	5.0	96.1	9.0
Bis(2-chloroethoxy)methane	5.0	92.4	9.8
Bis(2-chloroethyl)ether	5.0	96.0	11
Bis(2-chloroisopropyl)ether	5.0	95.2	12
Bis(2-ethylhexyl)phthalate	5.0	116	9.3
4-Bromophenylphenyl ether	5.0	108	9.0
Butyl benzyl phthalate	5.0	116	9.8
4-Chloroaniline	5.0	97.0	9.2
1-Chloronaphthalene	5.0	104	12
2-Chloronaphthalene	5.0	91.8	7.3
4-Chloro-3-methylphenol	5.0	107	12
2-Chlorophenol	5.0	94.5	7.8
4-Chlorophenyl phenyl ether	5.0	106	9.7
Chrysene	5.0	111	8.8
Dibenzo(a,j)acridine	5.0	10.6	34
Dibenzo(a,h)anthracene	5.0	110	5.9
Dibenzofuran	5.0	98.8	9.9
Di-*n*-butyl phthalate	5.0	113	9.4
1,2-Dichlorobenzene	5.0	89.9	12
1,3-Dichlorobenzene	5.0	87.6	13
1,4-Dichlorobenzene	5.0	87.3	13
3,3-Dichlorobenzidine	5.0	96.8	12
2,4-Dichlorophenol	5.0	97.5	8.0
2,6-Dichlorophenol	5.0	93.1	12
Diethyl phthalate	5.0	111	8.0
Dimethylaminoazobenzene	5.0	116	11
7,12-Dimethylbenz(a)anthracene	5.0	128	7.0
αα-Dimethylphenethylamine	5.0	7.0	4.1
2,4-Dimenthylphenol	5.0	107	9.4
Dimethyl phthalate	5.0	106	8.4
4,6-Dinitro-2-methylphenol	5.0	57.6	9.3
2,4-Dinitrophenol	5.0	17.2	39
2,4-Dinitrotoluene	5.0	98.2	6.2
2,6-Dinitrotoluene	5.0	98.5	9.9
1,2-Diphenylhydrazine[B]	5.0	108	11
Di-*n*-octyl phthalate	5.0	117	12
Ethyl methanesulfonate	5.0	77.9	10
Fluoranthene	5.0	110	8.7
Fluorene	5.0	101	10
Hexachlorobenzene	5.0	108	8.9
Hexachlorobutadiene	5.0	89.5	11
Hexachlorocyclopentadiene	5.0	60.9	14
Hexachloroethane	5.0	83.7	13
Indeno(1,2,3-cd)pyrene	5.0	99.2	6.2
Isophorone	5.0	88.7	8.5
3-Methylcholanthrene	5.0	117	8.6
Methyl methanesulfonate	5.0	48.5	28
2-Methylnaphthalene	5.0	104	9.3
2-Methylphenol	5.0	95.1	8.5
4-Methylphenol	5.0	92.4	11
Naphthalene	5.0	95.0	12
1-Naphthylamine	5.0	57.8	8.7
2-Naphthylamine	5.0	73.5	9.0
2-Nitroaniline	5.0	100	7.7
3-Nitroaniline	5.0	96.8	8.5
4-Nitroaniline	5.0	99.0	8.5
Nitrobenzene	5.0	88.4	19
2-Nitrophenol	5.0	85.3	10
4-Nitrophenol	5.0	104	6.0
N-nitroso-di-*n*-butylamine	5.0	97.5	9.3
N-nitroso-di-*n*-propylamine	5.0	87.5	20
N-nitrosopiperidine	5.0	90.8	7.6
Pentachlorobenzene	5.0	101	9.1
Pentachloronitrobenzene	5.0	109	9.7
Pentachlorophenol	5.0	86.2	8.1
Phenacetin	5.0	97.0	12
Phenanthrene	5.0	109	8.5
Phenol	5.0	97.3	9.2
2-Picoline	5.0	7.7	30
Pronamid	5.0	120	11
Pyrene	5.0	113	8.4
1,2,4,5-Tetrachlorobenzene	5.0	91.2	8.6
2,3,4,6-Tetrachlorophenol	5.0	104	7.3
1,2,4-Trichlorobenzene	5.0	89.3	11
2,4,5-Trichlorophenol	5.0	95.1	12
2,3,6-Trichlorophenol	5.0	96.4	6.7
2-Fluorobipenyl	2.5	92.9	8.0
2-Fluorophenol	5.0	95.4	7.7
Nitrobenzene-d_5	2.5	92.2	9.8
Phenol-d_5	5.0	98.9	9.7
Terphenyl-d_{14}	2.5	112	10
2,4,6-Tribromophenol	5.0	92.3	7.7

[A] The topsoil was dry when spiked. The number of determinations was three. Determinations were made by gas chromatography-mass spectrometry. All recoveries were corrected for analyte losses incurred during blowdown evaporation of solvent to determine, specifically, recoveries by microwave extraction.

[B] Determined as azobenzene.

iTeh Standards (https://standards.iteh.ai) Document Preview ASTM D6010-96 https://standards.iteh.ai/catalog/standards/sist/1d99b2d4-e006-47bb-8d71-9454c3610153/astm-d6010-96

6. Apparatus

6.1 *Microwave Heating System*—A laboratory microwave heating system capable of delivering a minimum of 900 W of microwave energy. The system should be capable of 1 % power adjustments and 1-s time adjustments. The microwave

TABLE 2 Semivolatile Analyte Recovery from ERA Soil (Lot 324)[A]

Analyte	Certified Concentration mg/kg[B]	Average Recovery, %[C,D]	RSD, %
Anthracene	1.01	68.6	4.7
Benzo(a)anthracene	2.03	103	6.7
Bis(2-ethylhexyl)phthalate	7.12	150	11.2
Butyl benzyl phthalate	10.6	128	10.8
2-Chlorophenol	5.08	76.2	15.7
Chrysene	2.35	114	8.5
Dibenzofuran	6.79	88.8	1.9
2,4-Dinitrotoluene	5.0	83.0	4.2
Fluorene	6.06	72.1	1.0
Naphthalene	1.64	64.3	15.7
Pentachlorophenol	12.2	85.0	6.8
Phenanthrene	1.57	110	6.8
Pyrene	8.03	110	12.8
2,4,5-Trichlorophenol	7.99	96.9	1.3
2,4,6-Trichlorophenol	4.56	71.1	4.7
2-Fluorobiphenyl	6.0	102	8.8
2-Fluorophenol	20.0	99.5	14.1
Nitrobenzene-d_5	5.0	87.4	15.8
Phenol-d_5	20.0	96.0	13.1
Terphenyl-d_{14}	5.0	142	8.4
2,4,6-Tribromophenol	20.0	94.8	3.9

[A] This soil was obtained from Environmental Resources Associates (ERA) in Arvada, CO, and has been certified for the compounds listed in this table.

[B] Reported by ERA.

[C] The number of determinations was four. The recoveries are based on the ERA certified values.

[D] Recoveries corrected for analyte losses incurred during blowdown evaporation of solvent to determine, specifically, recoveries by microwave extraction.