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.

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.

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.

TABLE 3 Chlorinated Pesticide Recovery from Freshly Spiked Topsoil[A]

Pesticide	Spike Level, μg/kg	Average Recovery, %[A]	RSD, %
Alachlor	200	87.6	2.3
Aldrin	20	87.0	2.1
α-BHC	20	94.4	4.1
β-BHC	20	90.4	3.6
γ-BHC	20	89.6	1.3
δ-BHC	20	96.9	2.8
Captafol	200	122	4.7
Captan	200	105	1.7
Chlorobenzilate	100	82.6	5.6
α-Chlordane	20	80.0	3.9
γ-Chlordane	20	86.2	2.9
Chloroneb	100	69.6	2.4
Chloropropylate	100	86.0	5.6
Chlorothalonil	60	83.4	0.9
DBCP	100	55.1	19.8
DCPA	60	93.3	1.5
4,4′-DDD	20	76.9	4.5
4,4′-DDE	20	84.7	3.1
4,4′-DDT	20	116	5.6
Diallate	200	98.6	4.1
Dichlone	200	77.7	2.6
Dichloran	60	110	9.3
Dieldrin	20	85.9	3.8
Endosulfan I	20	86.8	3.1
Endosulfan Ii	20	71.9	6.3
Endosulfan sulfate	20	81.6	6.5
Endrin	20	97.4	1.9
Endrin aldehyde	20	78.3	3.5
Endrin ketone	20	90.6	3.0
Etridiazole	100	92.6	14
Heptachlor	20	110	1.4
Heptachlor epoxide	20	95.3	2.7
Hexachlorobenzene	200	80.8	1.6
Hexachlorocyclopentadiene	100	107	12
Isodrin	20	103	1.3
Methoxychlor	60	90.9	2.7
Mirex	20	93.5	3.4
Nitrofen	100	90.2	3.6
PCNB	100	94.5	1.8
Perthane	200	86.8	4.0
Propachlor	100	91.5	8.4
Trans-Nonachlor	20	81.7	5.4
Cis-Permethrin	60	81.2	4.8
Trans-Permethrin	60	103	32
Trifluralin	40	101	2.4

[A] The number of determinations was three. The recoveries were corrected for analyte losses incurred during blowdown evaporation of solvent to determine, specifically, recoveries by microwave extraction.

TABLE 4 Organophosphorus Pesticide and Industrial Chemical Compound Recovery from Freshly Spiked Topsoil

Pesticide	Spike Level mg/kg	Average Recovery, %[A]	RSD, %
Aspon	1.0	91.8	3.4
Azinphos-methyl	2.0	103	0.9
Azinphos-ethyl	1.0	122	1.4
Bolstar	1.0	103	1.8
Chlorofenvinphos	2.0	1.02	3.8
Chlorpyrifos	1.0	90.9	3.8
Chlorpyrifos methyl	1.0	95.5	3.6
Coumaphos	2.0	100	0.7
Crotoxyphos	2.0	109	0.8
Demeton-O	1.0	98.7	7.1
Demeton-S	1.0	95.2	7.3
Dichlorofenthion	1.0	73.5	9.6
Dichlorvos	1.0	63.4	14
Dicrotophos	1.0	79.5	2.5
Dimethoate	1.0	85.8	8.8
Dioxathon	2.0	99.0	1.7
Disulfoton	1.0	97.5	3.5
EPN	1.0	102	1.3
Ethion	1.0	102	2.4
Ethoprop	1.0	106	3.6
Famphur	1.0	101	1.7
Fenithrothion	1.0	119	1.1
Fensulfothion	1.0	75.7	34
Fenthion	1.0	103	1.8
Fonophos	1.0	102	3.3
Leptophos	1.0	95.9	2.9
Malathion	1.0	105	1.1
Merphos	1.0	115	0.6
Mevinphos	2.0	66.1	17
Monochrotophos	1.0	9.4	8.3
Naled	2.0	9.4	15
Parathion ethyl	1.0	98.0	3.8
Phorate	1.0	94.4	4.9
Phosmet	1.0	99.1	0.8
Phosphamidon	2.0	16.6	29
Ronnel	1.0	99.5	2.9
Stirophos	2.0	99.0	2.7
Sulfotepp	1.0	93.0	4.3
TEPP	2.0	5.4	13
Thionazin	0.5	104	2.1
Tokuthion	1.0	99.0	3.0
Trichlorfon	1.0	7.8	16
Trichloronate	1.0	96.9	1.7
Industrial Chemicals			
Hexamethylphosphoramide (HMPA)	0.5	5.6	32
Tri-*o*-cresylphosphate (TOCP)	1.0	100	1.6
Triazine Herbicides			
Atrazine	1.0	100	0.9
Simazine	1.0	123	0.8

[A] The number of determinations was three. The analyte recoveries were corrected for losses incurred during blowdown evaporation of solvent to determine specificly recoveries by microwave extraction.

unit must be capable of measuring and controlling solvent temperature within an extraction vessel. The microwave cavity should be constructed so as to prevent any possible metal to metal arcing from occurring within the cavity. The oven cavity should be equipped with an exhaust ventilation sufficient to provide ten chamber exchanges/min. The ventilation exhaust should contain an air flow sensor and a solvent sensor capable of detecting no air flow and solvent concentrations below their lower explosive limits and shutting the microwave source off. The cavity shall have a 360° oscillating turntable to ensure even sample heating and be capable of removing contained vessel-vented solvents. Safety interlocks to shut off magnetron power output shall be contained in the cavity door opening mechanism. The system shall comply with the Department of Health and Human Services Standards under the CFR, Part 1030.10, Subpart (c)(1), (c)(2), and (c)(3), for microwave leakage. The system should have Federal Communication Commission (FCC) type approval for operations under FCC Rule Part 18. If the system is used outside of the United States it should comply with health and human service standards for the country in which it is used.

6.2 *Extraction Vessels*—A closed lined extraction vessel of approximately 100-mL capacity. The vessel consists of a fluoropolymer inner liner and cover to contain and isolate a solvent solution from a higher strength polymer outer pressure vessel body. The vessel assembly shall be transparent to microwave energy and capable of withstanding an internal pressure of 200 psi (13.8×10^6 dynes/cm^2) and a temperature of 200°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible vessel rupture and be connected to a sealed venting system capable of removing vented solvent vapors from the microwave cavity.

TABLE 5 PCB Recovery from Soils

Sample	Certified Value or Spike Level, mg/kg	Aroclor Type	Average Recovery, %[A]	RSD, %
Environmental Resource Associates soil	394[B]	1260	89.9[C]	2.6
Freshly spiked topsoil	0.100[D]	1016	85.9[E]	5.6
		1260	82.5[E]	4.2

[A] Analyte recoveries were corrected for losses incurred during blowdown evaporation of solvent to determine specificly recoveries by microwave extraction.
[B] Certified by the Environmental Resource Associates (ERA).
[C] The number of determinations was four.
[D] Spiked in the laboratory.
[E] The number of determinations was three.

6.3 *Temperature-Pressure Control Vessel*—A closed lined extraction vessel with temperature and pressure monitoring ports capable of withstanding an internal pressure of 200 psi (13.8×10^6 dynes/cm^2) and a temperature of 200°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible vessel rupture and that is connected to a sealed venting system capable of removing vented solvent vapors from the microwave cavity.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all extractions. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6]

7.2 *Acetone*, pesticide quality or equivalent.

7.3 *Hexane*, pesticide quality or equivalent.

7.4 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III of Specification D 1193.

8. Hazards

8.1 The microwave system shall be operated and maintained in accordance with the manufacturer's recommended safety precautions.

8.2 The extraction vessels shall be used and maintained in accordance with the manufacturer's recommended safety precautions.

8.3 Use only microwave systems and vessels approved for solvent use.

9. Sampling

9.1 Collect a soil, sediment, sludge, or waste sample using an appropriate sampling technique.

9.2 Prepare the soil, sediment, sludge, or waste sample in accordance with Practice D 3976, Test Method D 5368, and EPA Test Methods for Evaluating Solid Waste, Vol 1A.

9.3 *Determination of Water Content*—In certain cases, sample results are to be reported on a dry-weight basis. When such data are desired, a sample portion for water content determination should be weighed out at the same time as the portion used for organic analytical determination. Water content then is determined on that portion of the sample in accordance with Practice D 3976.

10. Vessel Cleaning

10.1 Follow the manufacturer's recommended cleaning procedure or clean the vessel liners and covers as follows:

10.1.1 Clean each liner and cover as soon as possible after use by rinsing with acetone-hexane (1 + 1), followed by washing with hot water and detergent, followed by rinsing with tap water and then with organic-free reagent water. Rinse with acetone-hexane (1 + 1), dry and store in a clean dust-free environment.

11. Procedure

11.1 Take a 5-g portion of the sample prepared in 9.2, weigh to the nearest 0.1 mg, and transfer into extraction vessels. Include an empty extraction vessel in each set as a method blank. The temperature-pressure control vessel shall contain 5 g of sample material.

11.2 Add 30 mL of acetone-hexane (1 + 1) to each sample and blank extraction vessel.

11.3 Close each extraction vessel according to the manufacturer's recommended procedures.

11.4 Place the closed extraction vessel into the instrument turntable and assemble following the manufacturer's suggested procedure.

11.5 Place the turntable containing evenly spaced vessels into the oven cavity, and make the proper connections to the control vessel following the manufacturers recommended procedures.

NOTE 1—If samples have different water contents, it is recommended that dry samples be extracted separately from wet samples, since dry and wet sample-solvent suspensions will heat at different rates, and thereby reach different temperatures.

11.6 Heat the vessels according to the extraction heating parameters in Table 6.

NOTE 2—For less than 7 vessels, microwave power can be reduced to less than 100 %.

11.7 Allow the vessels to cool to room temperature.

11.8 Open the vessels, decant, and filter or centrifuge to separate the solvent from the sample for further processing and cleanup if necessary before performing analyte determination.

12. Data

12.1 Typical analyte recoveries are contained in Tables 1-5.

12.2 Each laboratory using this practice will operate a formal Quality Control Program. The minimum requirements of this program must consist of an initial demonstration of

TABLE 6 Extraction Heating Parameters

Microwave power	100 %
Vessels pressure	150 psi
Vessels temperature	115°C
Heating time at 115°C	10 min

---

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

laboratory capability and the ongoing analysis of method blanks, duplicate and spiked samples to evaluate, and document quality data.

13. Precision and Bias

13.1 It is the responsibility of the user to establish the precision and bias of the extraction method in conjunction with the analytical measuring method.

14. Keywords

14.1 extraction; microwave; organic compounds; safety; solvent; vessel

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (http://www.astm.org).