



Standard Practice for Closed Vessel Microwave Solvent Extraction of Organic Compounds from Solid Matrices¹

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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 Water²

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis³

D 5368 Test Method for the Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples⁴

2.2 Other Standards:

United States Environmental Protection Agency (USEPA),
Test Methods for Evaluating Solid Waste Volume 1A: Laboratory Manual Physical/Chemical Methods⁵

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.

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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.

Title 21, Code of Federal Regulations (CFR), Part 1030, and
Title 47, Part 18⁵

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 leaching, 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 interferences.

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.