



Standard Practice for Extraction of Solid Waste Samples for Chemical Analysis Using Soxhlet Extraction¹

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^{ε1} NOTE—Editorial changes were made to 11.4 in December 2008.

1. Scope

1.1 This practice describes standard procedures for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sediments, sludges, and granular wastes using Soxhlet extraction.

1.1.1 The sample must be suitable for being mixed with the sample drying agent, sodium sulfate or magnesium sulfate, to provide drying of all sample surfaces.

1.2 This practice, when used in conjunction with Test Method D 5368 is applicable to the determination of the total solvent extractable content (TSEC) of a soil, sediment, sludge, or granular solid waste and depends upon the solvent chosen for extraction.

1.3 This practice is limited to solvents having boiling points below the boiling point of water at ambient pressure.

1.4 The solvent extract obtained by this practice may be analyzed for total or specific nonvolatile and semivolatile organic compounds but may require sample clean-up procedures prior to specific compound analysis.

1.4.1 This practice provides sample extracts suitable for analysis by various techniques such as gas chromatography with flame ionization detection (GC/FID) or gas chromatography with mass spectrometric detection (GC/MS).

1.5 This practice is recommended only for solid samples that can pass through a 10-mesh sieve (approximately 2-mm openings), or are less than 2 mm in thickness.

1.6 *This standard does not purport to address all of the safety problems, 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.* For specific precautions see Section 9.

¹ This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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2. Referenced Documents

2.1 ASTM Standards:²

D 75 Practice for Sampling Aggregates

D 420 Guide to Site Characterization for Engineering Design and Construction Purposes

D 2234/D 2234M Practice for Collection of a Gross Sample of Coal

D 2910 Method for Removal of Organic Matter from Water by Activated Carbon Absorption³

D 3086 Test Method for Organochlorine Pesticides in Water³

D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents

D 3975 Practice for Development and Use (Preparation) of Samples for Collaborative Testing of Methods for Analysis of Sediments

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis

D 4281 Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination

D 5368 Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples

2.2 EPA Document:

SW 846 Method 3540 Soxhlet Extraction, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW 846, Third Edition⁴

3. Terminology

3.1 Definitions:

3.1.1 GC—gas chromatography.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

3.1.2 *GC/MS*—gas chromatography with mass spectrometric detection.

3.1.3 *TSEC*—total solvent extractable content. The total concentration by weight (w/w) of organic materials that are extractable from a soil or solid waste by the selected solvent.

4. Summary of Practice

4.1 The sample is mixed with sodium sulfate or magnesium sulfate, placed in an extraction thimble, and extracted using an appropriate solvent in a Soxhlet extractor for a 15 to 20-h period. For additional information, see Test Method **D 4281**. The time required may be longer or shorter than the stated period, provided the extraction time selected has been demonstrated appropriate for the compounds and matrix of interest.

4.2 The solvent extract may be further processed using sample clean-up techniques and may be analyzed using instrumental methods for specific nonvolatile or semivolatile organic compounds. This practice does not include sample extract clean-up methods.

5. Significance and Use

5.1 This practice provides a general procedure for the solvent extraction of organics from soils, sediments, sludges, and fine-grained solid wastes. It may be used as the initial step in the solvent extraction of organic constituents from waste materials for the purpose of quantifying extractable organic compounds. When the appropriate extraction solvent is used, the procedure can be applied to the determination of the total solvent extractable content (TSEC) of the sample. The extraction of nonvolatile or semivolatile organic compounds from the

above solid waste should use solvents listed in **Table 1** or SW846 Method 3540.

5.2 The detection limit, linear concentration range, and sensitivity of the method for specific organic compound analysis will depend upon the method used for instrumental analysis and will also depend upon the sample clean-up and solvent concentration methods used. Typical detection limits that can be achieved for GC or GC/MS are in the parts per million and sub-parts per million range.

5.2.1 The method detection limit can be adjusted by varying the volume of extract used and by additional sample clean-up prior to analysis.

5.3 Soxhlet extraction has an advantage when analyzing solid waste and soil/waste mixtures which form emulsions with more rigorous solvent mixing extraction techniques.

6. Interferences

6.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts or elevated baselines which cause misinterpretation of follow-up analyses. All of these materials must be demonstrated to be free of interference under the conditions of the follow-up analysis. Specific selection of reagents or the purification of solvents by distillation in all glass systems, or both, are required when organic component analysis follows extraction.

6.1.1 Glassware should be cleaned by washing with detergent or non-chromate cleaning solution, rinsing first with tap water, then reagent water, then redistilled acetone, and finally with pesticide quality solvent (that is, the solvent used for extraction). If the type and size of glassware permits, it may be

TABLE 1 Selected Applications of Soxhlet Extraction for Extraction of Organic Constituents from Solid Matrices

Sample Matrix	Solvent	Compounds or Constituents	Extraction Time, h (cycles)	Reference
(1) Sediment	1,1,1-trichloro-1,2,2-trifluoroethane (Freon)	oil and grease	4 (80)	(1)^A Plumb (1983)
(2) Sludges and similar materials	1,1,1-trichloro-1,2,2-trifluoroethane (Freon)	oil and grease	4 (80)	(2) Standard Methods
(3) Sludges from sewage	hexane then methanol	total organic C oil, grease, fats	24	(3) Strachan (1983)
(4) Municipal wastewater suspended solids and activated carbon	hexane/dichloromethane	semivolatile priority pollutants	24 (480)	(4) Harrold (1982)
(5) Soil and housedust	acetone/hexane(1:1)	organochlorine insecticides	5 (60)	(5) EPA (1980)
(6) Sediment	dichloromethane	phenols	8	(6) Goldberg (1980)
(7) Soil	a) acetone/n-hexane(1:1) b) acetonitrile	aldrin, dieldrin	12 (554) 14 (47)	(7) Chiba (1968)
(8) Soil	c) 2-propanol/n-hexane(1:1) chloroform/methanol(1:1) (other solvents also studied)	aldrin, dieldrin	18 (108) 8 (160)	(8) Saha (1969)
(9) Airborne particulates	methanol (cyclohexane also studied)	gross organics	2	(9) Hill (1977)
(10) Airborne particulates	benzene	selected PAHs	4–6	(10) Pierce (1975)
(11) Airborne particulates	numerous solvents studied	selected PAHs	6	(11) Stanley (1967)
(12) Coke oven aerosol particulates	benzene	selected PAHs	2 (18–20)	(12) Broddin (1977)
(13) Artificial aerosol particulates	methanol/benzene methanol/benzene methanol/benzene	selected PAHs selected phthalates selected aliphatics	8 (80) 16 (160) 2 (20)	(13) Cautreels (1976)
(14) Activated carbon	methanol benzene chloroform	selected nitrogen aromatics selected nitrogen aromatics phenols	4 (40) 2 (20) 44 (440)	(14) Pahl (1973)
(15) Glass fiber filters	chloroform/ethanol	gross organics		(15) Buelow (1973)
(16) Surface sediments	26 solvents and 24 binary mixtures	total organic carbon	6	(16) Grosjean (1975)
(17) Bottom sediment	methanol then dichloromethane	total oil hydrocarbon	48 (160)	(17) Sporstol (1985)
(18) Environmental particulates	hexane/acetone/isooctane	chlorinated benzenes	18	(18) Onuska (1985)
(19) Soils	benzene	chlorinated dioxins	16	(19) Lamparski (1980)
	hexane/acetone/methanol	DDT	12	(20) Nash (1972)

^A The boldface numbers in parentheses refer to the list of references at the end of this practice.

cleaned by heating in a muffle furnace at 400°C for 15 to 30 min. Alternatively, glassware may be oven dried at 103°C for at least 1 h, after solvent rinsing and draining. Volumetric glassware should not be heated in a muffle furnace.

6.1.2 Plastics, except PTFE-fluorocarbon, can be a source of serious interference, especially when specific organic constituents are of analytical interest. Their use must be avoided. Samples should be collected in glass bottles with PTFE-fluorocarbon-lined caps. Alternatively, caps may be lined with precleaned aluminum foil when the pH of the sample is near neutral and the salt content of the sample is low. To minimize the possibility of contamination of containers after cleaning, these items should be cleaned immediately prior to use and capped. A glassware rinse should be performed with the extraction solvent immediately prior to use.

6.2 A blank Soxhlet extraction and analysis should be performed in order to determine the solvent, drying agent, and apparatus background. If necessary, the glass (or paper) thimble and glass wool should be leached with solvent prior to use in order to minimize contamination. Method blanks should be less than 20 % of the concentration of the minimum needed reportable concentration. A method blank(s) must be reported with method data.

6.3 A relatively high TSEC background (>20 % of the minimum TSEC of interest) can result from solubility of sodium sulfate or other drying agent in the extraction solvent. This will preclude the application of the method for TSEC determinations.

6.4 Impurities in the extracting solvent can be a source of interference or TSEC background. Solvent blanks should be analyzed with each new bottle of solvent. Whenever a high TSEC background, or interfering compounds, are traced to the solvent, a new source of solvent should be obtained. Alternatively, impurities can often be removed by distillation in glass.

6.5 If organic compound interferences are found in the anhydrous sodium sulfate or magnesium sulfate, the drying agent may be rinsed with pure extraction solvent, or alternatively, Soxhlet extracted, followed by oven drying.

6.6 Inorganic constituents extractable from the waste by the extraction solvent will result in a positive interference in the determination of TSEC. This potential for interference must be determined by the analyst on a case-by-case basis.

6.7 When specific organic compound analysis is of interest, matrix interferences may be caused by compounds that are coextracted from the sample (but are not of interest). The extent of such matrix interferences will vary considerably depending on the sample and the specific instrumental analysis method used. Matrix interferences may be reduced by a judicious choice of extracting solvent, or alternatively, by using a clean-up technique on the extract following Soxhlet extraction.

7. Selection of the Extraction Solvent

7.1 The selection of solvent for extraction will depend upon many factors, including the following (see [Table 1](#) for selected applications):

7.1.1 Boiling point of the solvent.

7.1.2 Boiling point of the compounds or class of compounds of interest.

7.1.3 Tendency of the solvent and matrix to form emulsions.

7.1.4 Solvent strength (that is, polarity, solubility of compounds of interest).

7.1.5 Safety of solvent use (that is, toxicity, flammability).

7.1.6 Purity of solvent.

7.1.7 Solvent compatibility with analytical instrumentation.

7.2 The analyst should demonstrate a recovery using a spiking procedure in the matrix of interest before using this procedure.

7.3 Because the extraction is carried out at temperatures approaching the boiling point of the solvent for the entire extraction period, the analyst must ensure that heat-labile and more volatile solutes that may be of interest are stable and recoverable by the method. Low-boiling fractions can also be lost in the solvent evaporation steps where Kuderna-Danish solvent concentration is performed or where TSEC is determined gravimetrically after solvent evaporation.

7.4 The rate and extraction efficiency of the Soxhlet extraction process are not only a function of the solubility of the constituents of interest in solvent versus the matrix, but are also related to the dissolution process. This depends upon the ease of penetration of the solvent into the solid sample matrix. Fine particles are extracted more easily than large particles because of the increased surface area they provide. Therefore the particle nature of the sample must be evaluated and documented.

7.5 In many cases where the extraction efficiency of the constituent of interest during the extraction period is less than 100 %, the efficiency of the solvent extraction process is highly dependent upon the control of conditions during the Soxhlet extraction process. Extraction efficiency will depend upon the combined effect of the specific solvent used, the temperature at which extraction occurs, the cycle time for the Soxhlet apparatus, and the total extraction time. Therefore the rate and duration of extraction must be closely controlled.

8. Apparatus

8.1 Soxhlet extraction apparatus consisting of Soxhlet extractor, Allihn condenser, and 500-mL round-bottom boiling flask.

8.1.1 *Allihn Condenser*, bottom standard taper joint 45/50.

8.1.2 *Boiling Flask*, flat bottom, standard taper joint 24/40.

8.1.3 *Soxhlet Extractor*, 85-mL, top standard taper joint 45/50, bottom standard taper joint 24/40.

8.2 *Glass or Paper Thimble or Glass Wool* to retain sample in Soxhlet extraction device. It should drain freely and may require cleaning before use. To clean the thimbles, pre-extract them with the solvent to be used for sample extraction.

8.3 *Boiling Chips, Glass Beads, or PTFE-fluorocarbon Boiling Stones*, approximately 10/40 mesh. Boiling chips or glass beads can be cleaned by heating to 400°C for 30 min. Alternatively, Soxhlet extract with the same solvent as will be used for sample extraction.

8.4 *Heating Source*, capable of heating Soxhlet apparatus to achieve 10 solvent cycles per hour. Most commercially available heating mantles are adequate.

8.5 *Kuderna-Danish Concentrator*, fitted with graduated evaporative concentrator tube.