



Standard Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples¹

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1. Scope

1.1 These test methods describe standard procedures for gravimetrically determining the total nonvolatile and semi-volatile organic content of solvent extracts from soils or solid wastes. The following methods are included:

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Method A—Micro-Determination of TSEC	11-13
Method B—Evaporating Dish Procedure	14-16
Method C—Boiling Flask Procedure	17-19

1.2 These methods are used after a solvent extract is obtained from a soil or solid waste. For these methods to be applicable, the extraction solvent must have a boiling point less than that of water at ambient pressure.

1.3 The total solvent extractable content (TSEC) of a soil, sediment, sludge, or solid waste depends upon the solvent and method used for the extraction procedure.

2. Referenced Documents

2.1 ASTM Standards:

- D 2109 Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures²
- D 2910 Practice for Concentration and Recovery of Organic Matter from Water by Activated Carbon³
- D 3086 Test Method for Organochlorine Pesticides in Water³
- D 3445 Test Method for Nonvolatile Matter in Trichlorotrifluoroethane⁴
- D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents³
- D 4281 Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination³
- D 5369 Practice for the Extraction of Solid Waste Samples for Chemical Analysis Using Soxhlet Extraction⁵

3. Terminology

3.1 Definition:

¹ These test methods are under the jurisdiction of ASTM Committee D34 on Waste Management and are the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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² *Annual Book of ASTM Standards*, Vol 15.05.

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

⁴ *Discontinued 1991*; Replaced by Test Methods D 2109, *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *Annual Book of ASTM Standards*, Vol 11.04.

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

4. Summary of Methods

4.1 The sample is extracted with an organic solvent using a procedure such as described in Practices D 5369 (Soxhlet Extraction) or Test Method D 4281. The quantity of material extracted into the solvent is determined as the residue weight after solvent evaporation. Similar ASTM methods are Test Methods D 2109 and D 3445, and Practice D 2910.

4.1.1 *Method A, Micro-Determination Method*—The residue weight after solvent evaporation is determined for 200 μ L of solvent extract evaporated on an aluminum weighing dish. A heat lamp is used to effect evaporation.

4.1.2 *Method B, Evaporating Dish Procedure*—The residue weight after solvent evaporation is determined for 1.0 to 75.0 mL of solvent extract. Solvent is evaporated in an evaporating dish by heating at 5°C below the solvent boiling point. A stream of nitrogen over the surface of the solvent is used to accelerate evaporation.

4.1.3 *Method C, Boiling Flask Method*—The residue weight after solvent evaporation is determined for 100 to 300 mL of solvent evaporated in a boiling flask. Solvent is evaporated in a water bath at the solvent boiling point.

5. Significance and Use

5.1 The TSEC provides a quantitative measure of the total solvent extractable organic content of the solid waste in question. Based upon the TSEC, the extract may be analyzed or further processed (that is, further cleanup or solvent concentration) in preparation for analysis. Thus, the TSEC provides a quantitative measure for optimizing the extractable organic concentration prior to chemical analysis.

5.2 The TSEC of soil or waste material may be used as a quantitative measure for the screening and selection of samples for chemical analysis.

5.3 The TSEC may be useful as a simple and relatively inexpensive quantitative indicator of changes occurring in the total extractable organic content of soil or waste materials.

5.4 The detection limit of the TSEC depends upon the specific procedures employed. Typical detection limits in solvent extracts (that is, without solvent pre-concentration) for Method A are more than 1000 μ g/g. For Method B typical

detection limits are 20 to 100 µg/g in the solvent extract. For Method C the typical detection limits are 3 to 20 µg/g for solutes in the solvent extract.

5.5 The sensitivity of the methods can be adjusted by varying the volume of solvent extract prior to gravimetric residue determination.

6. Interferences

6.1 Solvents, reagents, glassware, and other sample processing hardware may yield an unacceptable background relative to the limit of detection required for the TSEC measurement. A method blank must be performed in order to demonstrate the viability of the solvent and equipment used. Specific selection of reagents and the purification of solvents by distillation in all-glass systems may be required when low levels (that is, <100 µg/g) of TSEC are of interest.

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). For additional information, see Practices D 3694. If the type and size of glassware permits, it may be 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. Extracted samples should be stored in glass bottles with PTFE-fluorocarbon-lined caps. Extraction solvent volumes should be recorded and containers sealed to prevent solvent evaporation or cross contamination. Extracts should be refrigerated above the freezing point of the solvent.

6.1.3 If required, rinse glassware and utensils with extraction solvent prior to use in order to remove interferences. The method blank should be less than 20 % of the minimum reportable concentration.

6.1.4 Impurities in the extracting solvent can be a source of interferences 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.

6.2 A relatively high TSEC background can also result from inorganic drying agents (that is, Na₂SO₄, MgSO₄, CuSO₄, CaCl₂, CaSO₄, K₂CO₃, KOH, BaO, CaO, H₂SO₄, P₂O₅) in the extracting solvent. A relatively high background of any drying agent used (>20 % of the minimum TSEC concentration of interest) in the solvent will preclude the application of the method for TSEC determinations in those cases. Residue ignition and inorganic analysis are suggested to evaluate this potential background.

6.3 Organic interferences can arise from the extraction of impurities from inorganic drying agents. If this is suspected, the interference may be removed by rinsing the drying agent with pure extraction solvent followed by oven drying of the inorganic drying agent.

6.4 Inorganic constituents extractable from the waste by the

solvent selected will result in a positive interference in the determination of TSEC, unless removed prior to TSEC determination. This potential for interference must be determined by the analyst on a case by case basis using existing data and best judgment.

7. Selection of the Extraction Solvent

7.1 The selection of solvent for extraction and TSEC determination 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, and

7.1.7 Solvent compatibility with analysis instrumentation.

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

7.3 The extract is exposed to temperatures approaching the boiling point of the solvent during the evaporation procedure. Consequently, one must ensure that heat-labile and more volatile solutes that may be of interest are stable and recoverable by the method and the solvent of choice. Because low-boiling fractions are lost in solvent removal, reproducible results can only be obtained by strict adherence to method details.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents must conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁶ Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 When low concentrations of TSEC are to be determined (that is, <100 µg/g in the waste) pesticide-grade solvents (that is, distilled in glass) are recommended. The solvent blank for TSEC and the specific constituents of interest must be below 20 % of the reportable lower limit for the analysis.

8.3 *Acetone* ((CH₃)₂CO), technical grade.

8.4 *Explosion-Proof Oven or Furnace*.

8.5 *Heating Mantles*.

9. Precautions

9.1 Some solvents (for example, benzene, chloroform, and carbon tetrachloride) are suspected human carcinogens and must be handled by approved methods.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analytical Standards for Laboratory U.K. Chemical," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."