



# Standard Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples<sup>1</sup>

This standard is issued under the fixed designation D 5368; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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 Admixtures<sup>2</sup>
- D 2910 Practice for Concentration and Recovery of Organic Matter from Water by Activated Carbon<sup>3</sup>
- D 3086 Test Method for Organochlorine Pesticides in Water<sup>3</sup>
- D 3445 Test Method for Nonvolatile Matter in Trichlorotrifluoroethane<sup>4</sup>
- D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents<sup>3</sup>
- D 4281 Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination<sup>3</sup>
- D 5369 Practice for the Extraction of Solid Waste Samples for Chemical Analysis Using Soxhlet Extraction<sup>5</sup>

## 3. Terminology

### 3.1 Definition:

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.02.

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

<sup>5</sup> *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  $\mu$ 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  $\mu$ 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, Na<sub>2</sub>SO<sub>4</sub>, MgSO<sub>4</sub>, CuSO<sub>4</sub>, CaCl<sub>2</sub>, CaSO<sub>4</sub>, K<sub>2</sub>CO<sub>3</sub>, KOH, BaO, CaO, H<sub>2</sub>SO<sub>4</sub>, P<sub>2</sub>O<sub>5</sub>) 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.<sup>6</sup> 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* ((CH<sub>3</sub>)<sub>2</sub>CO), 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.

<sup>6</sup> "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 "Analar Standards for Laboratory U.K. Chemical," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

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

<sup>A</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

9.2 Explosive peroxides tend to form in ether solvent. A convenient means of testing for their presence is with E. M. Quant test paper.<sup>7</sup> This test should be performed before evaporation of any ether-bearing extract.

9.3 The use of fume hoods with volatile and toxic solvents is mandatory.

9.4 Flammable solvents must be protected from heat, sparks, or flames. Avoid buildup of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters. Keep containers closed. Use with adequate ventilation. Store bulk solvents in safety cabinets. Remove only a one-day supply and keep it in a hood.

9.5 Avoid prolonged breathing of vapor or spray mist and avoid prolonged or repeated skin contact for all organic solvents. Consult Material Safety Data Sheets for recommended handling procedures and precautions.

9.6 Samples containing odorous, known or suspected volatile materials, must be processed in a fume hood.

9.7 Samples known or suspected of containing toxic or hazardous materials must be handled in a fume hood. Safety information relative to the handling of any known toxic materials must be obtained and implemented prior to any sample handling.

## 10. Sample and Sample Preparation

10.1 Remove refrigerated solvent extract sample from storage and allow it to reach room temperature. Keep sample

sealed to prevent evaporation.

10.2 When sample has reached room temperature examine to ensure that the sample is homogeneous and does not contain insoluble matter. If necessary, remix and homogenize.

10.3 Record sample identification information for the solvent extract. Record or measure, if necessary, the solvent volume of the extract to be analyzed. Note any changes in the extract volume from the volumetric mark, if available, on the storage vial or container.

### Method A—Micro-Determination of TSEC

#### 11. Apparatus

11.1 *Microsyringe*, 250- $\mu$ L, or alternatively, a calibrated microdispenser capable of 200- $\mu$ L delivery. The delivery precision should be to 1 %.

11.2 *Aluminum Foil Weighing Pans, Micro*, approximately 1 to 6-cm diameter. Purchase or fabricate from oil-free aluminum foil.

11.3 *Analytical Semimicro Balance*, capable of reading to 0.00001 g.

11.4 *Balance Table*—An electronic load-cell type balance is recommended.

11.5 *Heating Lamp*, 250-W reflector-type bulb in a heat-resistant fixture whose height above the sample may be conveniently adjusted.

11.6 *Forceps*, 3 to 5-in., steel or nickel-plated steel.

11.7 *Desiccator with Desiccant*.

#### 12. Procedure

12.1 Dry an aluminum weighing dish for 1 h at  $105 \pm 5^\circ\text{C}$ . Cool in a desiccator for 30 min. Remove the weighing dish and

<sup>7</sup> E. M. Quant test paper, trademark of, and available from, MCB Manufacturing Chemists, Inc., 2909 Highland Avenue, Cincinnati, OH 45212 has been found satisfactory for this purpose.

allow it to equilibrate in ambient air for 30 min. Continue air equilibration until a constant weight is obtained.

12.2 With a 250- $\mu$ L syringe or microdispenser accurately transfer 200  $\mu$ L of solvent extract to a tared (or weighed) aluminum weighing dish.

12.3 Place the weighing dish under a heat lamp at a distance of 8 cm from the lamp for 1 min, or until dry, to allow the solvent to evaporate.

12.4 Place the weighing dish in a desiccator for 30 min, remove, and weigh to constant weight.

12.5 If the residue weight of the 200- $\mu$ L solvent extract aliquot is less than 0.00010 g, then a larger volume of solvent extract must be used for the TSEC determination by the application of Methods B or C.

### 13. Calculation

13.1 For TSEC of the sample, where no solvent pre-concentration was performed, calculate the TSEC as micrograms of residue per gram of sample ( $\mu$ g/g) using the following equation:

$$\text{TSEC} = \frac{(R_w)(V_e)(10^6)}{(S_w)(V_r)} \quad (1)$$

where:

TSEC = total solvent extractable content,  $\mu$ g/g,

$R_w$  = residue weight, g (that is, difference in weight of the tared aluminum weighing dish and the dish weight after solvent evaporation),

$S_w$  = weight of sample that was solvent extracted, g,

$V_e$  = total volume of solvent extract, mL, and

$V_r$  = volume of solvent evaporated in the determination of the residue weight, mL (that is, 200  $\mu$  L).

### Method B—Evaporating Dish Procedure

#### 14. Apparatus

14.1 *Evaporating Dish*, 125-mL capacity, platinum, porcelain, or high-silica glass, or glass beaker of similar capacity.

14.2 *Oven*, thermostatically controlled at  $105 \pm 5^\circ\text{C}$ .

14.3 *Analytical Balance*, capable of reading to 0.0001 g ( $\pm 0.0002$  g). An electronic load-cell type balance is recommended.

14.4 *Temperature-Controlled Water Bath* ( $\pm 2^\circ\text{C}$ ) or *Temperature-Controlled Hot Plate*—Use water bath or hot plate in a laboratory fume hood.

14.5 *Metal Tongs, Evaporating Dish*, safety, 9/2 in.

14.6 *Glass-Transfer Pipets*, 1, 10, 25, 50, and 75-mL volume.

14.7 *Desiccator with Desiccant*.

#### 15. Procedure

15.1 Dry a 125-mL evaporating dish, or glass beaker, for 1 h at  $105^\circ\text{C}$ . Wipe the outside of the dish or beaker with a lint-free cloth. Cool in a desiccator for 1 h, remove the dish or beaker, and weigh to constant weight.

15.2 Transfer an accurately measured volume of solvent extract (1.0 to 75.0 mL) to the evaporating dish, or beaker, using a pipet.

15.3 Place the weighing dish, or beaker, on the water bath or on a warm plate with the temperature set at  $5 \pm 2^\circ\text{C}$  below the

boiling point of the extracting solvent. Accelerate solvent evaporation by passing a gentle stream of nitrogen over the sample by means of a disposable Pasteur pipet.<sup>8</sup> Glass, metal, or PTFE tubing and glass pipets should be used for the nitrogen, and a new pipet should be used for each sample.

15.4 When the solvent has evaporated remove the dish, or beaker, and contents from the water bath (or from the warm plate) with metal tongs. Wipe the outside of the dish, or beaker, with lint-free cloth and a small amount of acetone to remove any water adhering to the dish. If a warm plate is used to heat the sample, the dish wipe step is unnecessary.

15.5 Place the weighing dish in a desiccator for 1 h, remove, and weigh immediately to constant weight.

### 16. Calculation

16.1 Calculate the TSEC as micrograms of residue per gram of sample ( $\mu$ g/g) using the following equation:

$$\text{TSEC} = \frac{(R_w)(V_e)(10^6)}{(S_w)(V_r)} \quad (2)$$

where:

TSEC = total solvent extractable content,  $\mu$ g/g,

$R_w$  = residue weight, g (that is, difference in weight of the tared evaporating dish and the dish weight after solvent evaporation),

$S_w$  = weight of sample that was solvent extracted, g,

$V_e$  = total volume of solvent extract, mL, and

$V_r$  = volume of solvent evaporated in the determination of the residue weight, mL.

### Method C—Boiling Flask Procedure

#### 17. Apparatus

17.1 *Oven*, thermostatically controlled at  $105 \pm 5^\circ\text{C}$ .

17.2 *Analytical Balance*, capable of weighing to 0.0001 g ( $\pm 0.0002$  g). An electronic load-cell type balance is recommended.

17.3 *Temperature-Controlled Water Bath* ( $\pm 2^\circ\text{C}$ ) or *Temperature-Controlled Hot Plate*—Use water bath or hot plate in a laboratory fume hood.

17.4 *Metal Tongs, Evaporating Dish*, safety, 9/2 in.

17.5 *Glass Pipets or Graduated Cylinders*, 100, 250, or 500-mL.

17.6 *Boiling Flask*, 250 or 500-mL.

17.7 *Desiccator with Desiccant*.

#### 18. Procedure

18.1 Dry a 250 or 500-mL boiling flask for 1 h at  $105 \pm 5^\circ\text{C}$ . Carefully wipe the outside of the flask with a lint-free cloth. Cool in a desiccator for 1 h, remove, and weigh to constant weight.

18.2 Transfer an accurately measured volume (100 to 300 mL) of the solvent extract to the boiling flask by means of a pipet.

18.3 Evaporate the solvent from the boiling flask on a water bath or warm plate set at the boiling point temperature of the

<sup>8</sup> Refer to Test Method D 3086 under Alternative Method for Final Concentration.

extraction solvent. When only traces of solvent remain, leave the flask on the water bath and draw air through the flask using vacuum for 5 min to remove any remaining solvent vapor or residual water. Carefully wipe the exterior of the flask with a lint-free cloth and a small amount of acetone to remove any water adhering to the flask. If a warm plate is used to heat the sample, the dish wipe step is unnecessary.

18.4 Place in a desiccator for 1 h, remove, and weigh immediately to a constant weight.

## 19. Calculation

19.1 Calculate the TSEC as micrograms of residue per gram of sample ( $\mu\text{g/g}$ ) using the following equation:

$$\text{TSEC} = \frac{(R_w)(V_e)(10^6)}{(S_w)(V_r)} \quad (3)$$

where:

- TSEC = total solvent extractable content,  $\mu\text{g/g}$ ,  
 $R_w$  = residue weight, g (that is, difference between the tare weight of flask and the weight of the flask after removal of extraction solvent),  
 $S_w$  = weight of sample that was solvent extracted, g,  
 $V_e$  = total volume of solvent extract, mL, and  
 $V_r$  = volume of solvent evaporated in the determination of the residue weight, mL (that is, 100 to 300 mL).

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