



Designation: D 5369 – 93 (Reapproved 1998)

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

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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.

2. Referenced Documents

2.1 ASTM Standards:

D 75 Practices for Sampling Aggregates²

D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes³

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 *Annual Book of ASTM Standards*, Vol 04.03.

³ *Annual Book of ASTM Standards*, Vol 04.08.

D 2234 Test Method for Collection of a Gross Sample of Coal⁴

D 2910 Practice for Concentration and Recovery of Organic Matter from Water by Activated Carbon⁵

D 3086 Test Method for Organochlorine Pesticides in Water⁵

D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents⁵

D 3975 Practice for Preparation of Samples for Collaborative Testing of Methods for Analysis of Sediments⁵

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis⁵

D 4281 Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination⁵

D 5368 Test Method for the Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples⁶

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process⁷

2.2 EPA Document:

SW 846 Method 3540 Soxhlet Extraction, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW 846, Third Edition⁸

3. Terminology

3.1 Definitions:

3.1.1 *GC*—gas chromatography.

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.

⁴ *Annual Book of ASTM Standards*, Vol 05.05.

⁵ *Annual Book of ASTM Standards*, Vol 11.02.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ *Annual Book of ASTM Standards*, Vol 11.04.

⁸ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

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 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

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 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 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 methanol benzene	selected PAHs selected phthalates selected aliphatics selected nitrogen aromatics selected nitrogen aromatics	8 (80) 16 (160) 2 (20) 4 (40) 2 (20)	(13) Cautreels (1976)
(14) Activated carbon	chloroform chloroform/ethanol	phenols gross organics	44 (440)	(14) Pahl (1973) (15) Buelow (1973)
(15) Glass fiber filters	26 solvents and 24 binary mixtures	total organic carbon	6	(16) Grosjean (1975)
(16) Surface sediments	methanol then dichloromethane	total oil hydrocarbon	48 (160)	(17) Sporstol (1985)
(17) Bottom sediment	hexane/acetone/isooctane	chlorinated benzenes	18	(18) Onuska (1985)
(18) Environmental particulates	benzene	chlorinated dioxins	16	(19) Lamparski (1980)
(19) Soils	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.



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.

8.5.1 *Kuderna-Danish Concentrator Receiving Vessel*, graduated 10-mL. A ground glass stopper is used to prevent evaporation of extracts.

8.5.2 *Kuderna-Danish Evaporative Flask*, 500-mL, attached to the concentrator tube with springs.

8.5.3 *Kuderna-Danish Evaporative Flask*, 40-mL, attached to the concentrator tube with springs.

8.5.4 *Kuderna-Danish Snyder Column*, three-ball macro.

8.5.5 *Kuderna-Danish Snyder Column*, two-ball micro.

8.6 *Water Bath for Kuderna-Danish*, heated with concentric ring cover, capable of temperature control ($\pm 2^\circ\text{C}$). The bath must be used in a hood.

8.7 *Chromatographic Column*: borosilicate, 20-mm inside diameter, approximately 400 mm

long, with coarse fritted plate on bottom and an appropriate solid phase.

8.8 *Impermeable Sheet of PTFE-fluorocarbon-Coated Flexible Material*, approximately 2 ft², for sample mixing, quartering, and preparation.

8.9 *Stainless Steel Utensils Coated With PTFE-fluorocarbon*, or other appropriate material for sample handling and mixing (that is, spatula, trowel, scoop, etc.).

8.10 *Stainless Steel or Brass Sieve*, 10 mesh.

9. Reagents and Materials

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁹ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

9.2 The extraction solvent of choice should be appropriate for the matrix and compounds of interest. This choice is dependent upon the chemical properties of the organic constituents of interest and the matrix being extracted. Literature references for solvents that have been used for Soxhlet extractions are provided in Table 1.

9.3 When specific organic compounds are to be analyzed, pesticide grade solvents (distilled in glass) are recommended. In general, the solvent blank for TSEC or for the specific compound of interest should be less than 20 % of the lowest reportable concentration required for the analysis.

9.4 *Sodium Sulfate (Na₂SO₄)*, or *Magnesium Sulfate (MgSO₄)*, reagent grade, granular, anhydrous, prepared by heating at 400°C for a minimum of 4 h in a shallow tray to eliminate interfering organics.

10. Precautions

10.1 Some solvents (for example, benzene, chloroform, and carbon tetrachloride) are suspected human carcinogens and must be handled accordingly.

10.2 Explosive peroxides tend to form in ether solvents. A convenient means of testing for their presence is with E.M. Quant test paper.¹⁰

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

10.4 Flammable solvents must be kept 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.

10.5 Avoid prolonged breathing of vapor or spray mist and avoid prolonged or repeated skin contact with any organic

solvent. Consult Materials Safety Data Sheets for handling instructions and precautions.

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

10.7 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 taken into account prior to any sample handling.

11. Sample and Sample Preparation

11.1 Obtain a representative sample of the waste or soil to be tested using ASTM sample methods developed for the specific industry. (See Practices D 75, D 420, D 2910, D 3694, and D 3975, and Test Method D 2234.)

11.2 Where no specific methods are available, use sample methodology for materials of similar physical form.

11.3 Determine the minimum sample size for collection and delivery to the laboratory in accordance with Practice E 122.

11.4 Keep samples in closed containers appropriate to the sample type prior to testing. Store biologically active samples at 4°C and their extraction started within 8 h of receipt.

11.5 Document the physical description of the sample to be tested, including particle characteristics, as far as is known or can be determined by visual observation.

11.6 Do not use organic materials such as rubber or plastics other than clean PTFE-fluorocarbon in the storage or handling of samples.

11.7 Prepare sediment and soil samples in accordance with the instructions in Practice D 3976. This practice includes procedures for the determination of the moisture content of the sample, allowing final results to be reported on a wet or dry basis, as appropriate.

11.8 Samples may be screened to remove foreign objects prior to preparation or homogenization for chemical analysis. Large objects are removed mechanically.

11.9 For field-cored wastes or castings produced in the laboratory, cut a representative vertical section weighing approximately 100 g for extraction, plus any additional samples required for the determination of solids content or other physical properties. If the material cannot be ground into a fine granular consistency, then it is not appropriate for Soxhlet extraction.

11.10 For free-flowing particulate wastes, prepare a sample of the required size by quartering on a sheet of PTFE-fluorocarbon material or PTFE-fluorocarbon-coated material that has been demonstrated to be noncontaminating, as follows:

11.10.1 Empty the sample container onto the center of the sheet.

11.10.2 Flatten out the sample gently with a suitable straightedge until it is spread uniformly to a depth appropriate for its particle size.

11.10.3 Remix the sample by lifting a corner of the sheet and drawing it across to the opposite corner in a manner that the material is made to roll over and over and does not merely slide along. Continue operation with each corner, proceeding in a clockwise direction. Repeat this operation ten times.

11.10.4 Lift all four corners of the sheet towards the center and holding all four corners together, raise the entire sheet into

⁹ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analytical Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

¹⁰ Test paper, such as E.M. Quant, available from MCB Manufacturing Chemists, Inc., 2909 Highland Avenue, Cincinnati, OH 45212.

the air to form a pocket for the sample.

11.10.5 Repeat 11.10.2.

11.10.6 With a straightedge at least as long as the flattened mound of sample (such as a thin-edged yard stick) gently divide the sample into quarters.

11.10.7 Discard alternate quarters.

11.10.8 If further reduction of the sample size is necessary, repeat 11.10.3 through 11.10.7. A minimum sample size equivalent to 5 g, dry weight, is recommended for each Soxhlet extraction. The exact sample size used for Soxhlet extraction must be recorded.

11.11 *Sample Sieving*—The analytical sample for Soxhlet extraction is defined as that material which passes a 10-mesh sieve (approximately 2-mm openings). Stainless steel or brass sieves may be used. Materials that cannot be sieved but are of a thickness of less than 2 mm (that is, after drying in accordance with 12.3 and grinding in accordance with 12.4) or where the material is penetrable by the extracting solvent, can still be Soxhlet extracted. The material must be suitable for being mixed with the sample drying agent, sodium sulfate or magnesium sulfate, to provide drying of all sample surfaces.

11.11.1 If the purpose of extracting is to measure specific compounds, recovery experiments will have to be performed to demonstrate the efficacy of sample handling and sieving steps (or whether those steps should be omitted).

11.11.2 When appropriate, sieve dry samples without further pretreatment.

11.11.3 Vigorously stir wet samples to incorporate as much liquid as possible, thereby facilitating subsequent wet sieving.

11.11.4 Pass the wet sample, preferably as a slurry, through the sieve. The bottom of an appropriately sized Erlenmeyer flask may be used to gently press the waste sample through the sieve. Manually remove large objects that will not pass through the sieve and will interfere with the sieving operation. These objects are not considered appropriate for Soxhlet extraction.

11.11.5 Mix the sieved material by stirring, then allow it to settle for subsequent removal of the supernatant liquid. A centrifuge may be used to facilitate the separation of the liquid and solid.

12. Procedure

12.1 Decant any supernatant liquid from the sieved and settled sample. Save the supernatant liquid if separate analysis is required.

12.2 Blend 10 to 15 g of homogenized solid sample, “as received” from 12.1, with an equal weight of anhydrous sodium sulfate or magnesium sulfate in a 150-mL glass beaker. Add the drying agent slowly with continuous stirring and continue stirring until a dry powder or dry granules are formed.

12.3 Allow this mixture to stand 15 to 30 min until solidified. More drying agent may be added if needed to give the sample a dry appearance.

12.4 Remove the solidified material and grind it in a porcelain or glass mortar.

12.5 Place the ground sample in a thimble of appropriate size. The thimble should extend just above the siphon arm of the Soxhlet apparatus. Add sufficient sample blend so that at least 5 g of dry weight sample is extracted. If disintegration or floatation of sample occurs, cover with a plug of glass wool

which has been previously extracted with the solvent. The glass wool will also aid in distributing the condensed solvent over the sample. Place the thimble in the extractor.

12.6 Add 275 mL of the extraction solvent into a 500-mL round-bottom flask containing a boiling chip, glass beads, or PTFE-fluorocarbon balls and attach the flask to the extractor. Initiate water flow through the cooling condenser.

12.7 Gradually heat the solvent to its boiling point. The solvent vapors will condense and drip into the thimble. The rate of extraction is varied by adjusting the heat supplied to the heating mantle. When the extraction thimble has filled to the top of the side arm, siphon the solvent automatically and drain back into the boiling flask. Adjust the heat until there is one dischargeable cycle every six minutes (that is, ten cycles per hour).

12.8 Extract the sample for 15 to 20 h. The extraction time may be shorter or longer, provided that the time selected has been demonstrated to be appropriate for the extraction (at least 80 % of the concentration which is normally extracted during the 15 to 20-h extraction procedure in the matrix of interest).

12.9 Turn off the heating mantle and allow the extract to cool. Rinse the condenser with three portions of 5 mL each of the extraction solvent and drain the Soxhlet into a graduated 500-mL glass cylinder. Record the volume of solvent recovered.

12.10 Filter and dry the extract by passing it through a 20-mm inside diameter column containing 10 cm of sodium sulfate which has been prewashed with the extracting solvent. Collect the eluent in a graduated 500-mL glass cylinder. Wash the sodium sulfate column with 25 mL of extracting solvent and add it to the graduated cylinder. Dilute the extract to exactly 300 mL with solvent.

12.11 Using a portion of the solutions from 12.10, determine the TSEC of the sample using TSEC Method A, B, or C in accordance with Test Method D 5368.

12.12 If the residue weight of the extract is less than 500 µg/g (with respect to the original solid), concentrate the solvent extract using the Kuderna-Danish apparatus prior to any other analysis (see 12.13).

12.13 Add the solvent extract to a 500-mL Kuderna-Danish concentrator flask fitted with a 10-mL graduated receiving vessel. Add one or two clean boiling chips or PTFE-fluorocarbon balls to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL solvent to the top. Place the Kuderna-Danish apparatus on a steam bath or hot water bath in a fume hood so that the concentrator tube and the entire rounded surface of the flask are bathed in hot water or vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter, but the chamber will not flood with condensed solvent. When the apparent volume of liquid reaches 1 to 3 mL, remove the Kuderna-Danish apparatus and allow it to drain for at least 10 min while cooling.

12.14 Rinse the Kuderna-Danish apparatus with 2 to 4 mL of solvent. Remove the Snyder column and flask and adjust the sample volume to 10.0 mL with solvent.

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12.15 If desired, measure the TSEC of the sample using TSEC Method A or B in accordance with Test Method D 5368. Proceed with instrumental analysis or further sample cleanup if required.

12.16 If necessary to meet the sensitivity requirements for the instrumental method used, the extract may be further concentrated 10-fold in the Kuderna-Danish receiver vessel.

12.16.1 Suspend the graduated receiver vessel in a beaker of warm water, a heating bath, or a heating block at a temperature 10 to 20°C below the boiling point of the solvent. Aid solvent evaporation by passing a gentle stream of nitrogen gas (prepurified quality or better) over the extract by means of a

disposable Pasteur pipet (that is, nitrogen blowdown).¹¹ Use glass, metal, or PTFE-fluorocarbon tubing and glass pipets for the nitrogen, and use a new pipet for each sample.

12.16.2 When the extract has evaporated to a little less than 0.5 mL, remove the concentration vessel from the heating device and allow it to cool to room temperature. Rinse the vessel with about 0.5 mL of solvent and dilute to the 1.0-mL mark.

12.17 If required, determine the TSEC of the concentrated extract using Method A or B in accordance with Test Method D 5368.

12.18 Proceed with further analysis of the concentrate or with further sample cleanup procedures as appropriate.

¹¹ Refer to Test Method D 3086 under Alternative Method for Final Concentration.

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