Designation: D5765 – 16

Standard Practice for Solvent Extraction of Total Petroleum Hydrocarbons from Soils and Sediments Using Closed Vessel Microwave Heating[1]

This standard is issued under the fixed designation D5765; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the solvent extraction of total petroleum hydrocarbon (TPH) from soils and sediments, using closed vessel microwave heating, for subsequent determination by gravimetric or gas chromatographic techniques.

1.2 This practice is recommended only for solid samples that can pass through a ten mesh screen (approximately 2 mm openings).

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

1.4 This practice is limited to solvents that are recommended for use in microwave solvent extraction systems.

1.5 The values stated in SI units are to be regarded as standard.

1.5.1 *Exception*—The inch-pound values given for units of pressure are to be regarded as standard; SI unit conversions are shown in parentheses.

1.6 *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.* Specific hazard statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

D3974 Practices for Extraction of Trace Elements from Sediments

D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

D5368 Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples (Withdrawn 2014)[3]

2.2 *Federal Standard:*

Code of Federal Regulations Title 21, Part 1030; and Title 47, Part 18[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

4. Summary of Practice

4.1 The chemical portion of this practice involves solvent extraction to dissociate petroleum hydrocarbons from the matrix.

4.2 The sample is extracted with acetone/hexane in a sealed microwave transparent vessel using microwave heating to an internal temperature of 150°C.

4.3 This practice provides a sample suitable for analysis by gas chromatography or gravimetric measurements.

5. Significance and Use

5.1 Solvent extraction of soils and sediments can provide information on the availability of petroleum hydrocarbons to leaching, water quality changes, or other site conditions.

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

5.2 Rapid heating, in combination with temperatures in excess of the atmospheric boiling point of acetone/hexane, reduces sample preparation or extraction times.

5.3 Reduced amounts of solvents are required and solvent loss due to boiling and evaporation are eliminated by use of closed extraction vessels.

6. Interferences

6.1 No interferences to the extraction of soils and sediments using microwave heating have been observed.

6.2 Precautions should be exercised to avoid those interferences normally associated with the final determination of total petroleum hydrocarbons using gas chromatography or gravimetric techniques.

7. Apparatus

7.1 *Microwave Heating System*—A laboratory microwave heating system capable of delivering a minimum of 900 W of microwave energy. The system should be capable of 1 % power adjustments and 1 s time adjustment. The microwave cavity should be constructed so as to prevent any possible metal to metal arcing from occurring within the cavity. The oven cavity should be equipped with an exhaust ventilation sufficient to provide ten chamber exchanges per minute. The ventilation exhaust should contain a solvent sensor capable of detecting solvent concentrations below their lower explosive limits and shutting the microwave source off. The cavity shall have a 360° oscillating turntable to ensure even sample heating, and be capable of monitoring and control of solvent temperature using either an in-situ control vessel or by external vessel monitoring. Safety interlocks, to shut off magnetron power output shall be contained in the cavity door opening mechanism. The system shall comply with Department of Health and Human Services Standards under the Code of Federal Regulations, Part 1030.10, Subparts (c) (1), (c) (2), and (c) (3), for microwave leakage. The system should have Federal Communications Commission (FCC) type approval for operations under FCC Rule Part 18.

7.2 *Extraction Vessels*—A closed lined extraction vessel of approximately 50 to 100 mL capacity. The vessel consists of a fluoropolymer or glass inner liner and fluoropolymer cover to contain and isolate a solvent solution from a higher strength polymer outer pressure vessel body. The vessel assembly shall be transparent to microwave energy and capable of withstanding an internal pressure of 300 psi (20.68 by 10^6dynes/cm^2) and a temperature of 200°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible rupture.

7.3 *Temperature-Pressure Control Vessel*—A closed lined extraction vessel with temperature and pressure monitoring ports capable of withstanding an internal pressure of 300 psi (20.68 by 10^6 dynes/cm^2) and a temperature of 200°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible vessel rupture.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. [5]

8.2 *Acetone,* HPLC grade.

8.3 *Hexane,* HPLC grade.

8.4 *Sodium Sulfate*—Reagent grade, granular, anhydrous, prepared by heating at 400°C for a minimum of 4 h.

8.5 *Ashless Filter Paper.*

9. Hazards

9.1 The microwave system shall be operated and maintained in accordance with the manufacturer's recommended safety precautions.

9.2 The extraction vessels shall be used and maintained in accordance with the manufacturer's recommended safety precautions.

9.3 Use only microwave systems and vessels approved for solvent use.

10. Sampling

10.1 Collect a soil or sediment sample using an appropriate sampling technique.

10.2 Prepare the soil or sediment sample in accordance with Practices D3976, D3974, or D3694.

11. Vessel Cleaning

11.1 Follow the manufacturer's recommended cleaning procedure.

12. Procedure

12.1 Take a 5 g portion of the sample prepared in 10.2, weigh to the nearest 0.1 mg, and transfer into extraction vessels. Include an empty extraction vessel in each set as a method blank.

NOTE 1—The temperature-pressure control vessel shall contain 5 g of sample material.

12.2 Add 30 mL of acetone/hexane (1 + 1) to each sample and blank extraction vessel.

12.3 Close each extraction vessel according to the manufacturer's recommended procedures.

12.4 Place the closed extraction vessels into the instrument turntable and assemble following the manufacturer's suggested procedure.

12.5 Heat the vessels according to the extraction heating parameters in Table 1.

12.6 Allow the vessels to cool to room temperature.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Extraction Heating Parameters

Microwave power	100 %
Vessels pressure	200 psi
Vessels temperature	150°C
Heating time at 150°C	15 min

12.7 Open the vessels and add 5 g of sodium sulfate to the sample and solvent mixture and gently swirl the mixture.

12.8 Filter the extract mixture through ashless filter paper rinsing and washing the extracted, sample, sodium sulfate and filter paper thoroughly with (1 + 1) acetone/hexane into an appropriate container. (See Guide D3856.)

NOTE 2—For gravimetric TPH determination, filter extract solvent into an appropriate pre-weighed evaporating dish. (See Test Methods D5368.)

12.9 Proceed with an appropriate procedure for gravimetric or gas chromatographic TPH determination.

13. Data

13.1 Typical data are shown in Table 2.

TABLE 2 Total Petroleum Hydrocarbon Recovery Data

Material	TPH Present (%)	TPH Recovered (%)	Measurement Method
Soil	1.34 [A]	1.32	Gravimetric
Soil [B]	0.214	0.223	Gravimetric
Soil [B]	0.099	0.094	GC-FID

[A] Value determined by Soxhlet Extraction.
[B] Environmental Resource Associates standard reference soil.

14. Precision and Bias

14.1 It is the responsibility of the user to establish the precision and bias of the extraction practice in conjunction with the analytical measuring method.

15. Keywords

15.1 extraction; microwave; solvent; total petroleum hydrocarbon (TPH); vessel