



Standard Practice for Solvent Extraction of Total Petroleum Hydrocarbons from Soils and Sediments Using Closed Vessel Microwave Heating¹

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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 pounds per square inch (psi) are to be regarded as the standard. The SI units given in parentheses are for information only.

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 8.

2. Referenced Documents

2.1 ASTM Standards:

D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents²

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water³

D 3974 Practice for Extraction of Trace Elements from Sediments²

D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis²

D 5368 Test Method for the Gravimetric Determination of

Total Solvent Extractable Content (TSEC) of Solid Waste Samples⁴

2.2 Federal Standard:

Code of Federal Regulations, Title 21, Part 1030, and Title 47, Part 18⁵

3. Summary of Practice

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

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

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

4. Significance and Use

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

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

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

5. Interferences

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

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

6. Apparatus

6.1 *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 %

¹ This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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² *Annual Book of ASTM Standards*, Vol 11.02.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.04.

⁵ Available from the U.S. Government Printing Office, Washington, DC.

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 removing contained vessel vented solvents. 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.

6.2 *Extraction Vessels*—A closed lined extraction vessel of approximately 100 mL capacity. The vessel consists of a fluoropolymer inner liner and 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 400 psi (27.57 by 10⁶ dynes/cm²) and a temperature of 250°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible rupture and that is connected to a sealed venting system capable of removing vented solvent vapors from the microwave cavity.

6.3 *Temperature-Pressure Control Vessel*—A closed lined extraction vessel with temperature and pressure monitoring ports capable of withstanding an internal pressure of 400 psi (27.57 by 10⁶ dynes/cm²) and a temperature of 250°C. The vessel assembly shall contain a safety pressure relief device that will prevent possible vessel rupture and that is connected to a sealed venting system capable of removing vented solvent vapors from the microwave cavity.

7. Reagents and Materials

7.1 *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.⁶

7.2 *Acetone*, HPLC grade.

7.3 *Hexane*, HPLC grade.

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

7.5 *Ashless Filter Paper*.⁷

⁶ *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.

⁷ Whatman No. 40, available from Whatman Specialty Products, 6 Just Rd., Fairfield, NJ 07004, or equivalent, has been found satisfactory.

8. Hazards

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

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

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

9. Sampling

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

9.2 Prepare the soil or sediment sample in accordance with Practice D 3976, Practice D 3974, or Practices D 3694.

10. Vessel Cleaning

10.1 Follow the manufacturer's recommended cleaning procedure.

11. Procedure

11.1 Take a 5 g portion of the sample prepared in 9.2, 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.

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

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

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

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

11.6 Allow the vessels to cool to room temperature.

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

11.8 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 D 3856.)

NOTE 2—For gravimetric TPH determination, filter extract solvent into an appropriate pre-weighed evaporating dish. (See Test Method D 5368.)

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

12. Data

12.1 Typical data are shown in Table 2.

TABLE 1 Extraction Heating Parameters

| | |
|-----------------------|---------|
| Microwave power | 100 % |
| Vessels pressure | 200 psi |
| Vessels temperature | 150°C |
| Total heating time | 45 min |
| Heating time at 150°C | 15 min |

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 |

^AValue determined by Soxhlet Extraction.

^BEnvironmental Resource Associates standard reference soil.

13. Precision and Bias

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

14. Keywords

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

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