



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

This standard is issued under the fixed designation D 5765; 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 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 Practices

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 % power adjustments and 1-s time adjustment. The microwave

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.

Current edition approved Aug. 15, 1995. Published October 1995.

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.