



Designation: D 5258 – 02

Standard Practice for Acid-Extraction of Elements from Sediments Using Closed Vessel Microwave Heating¹

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1. Scope

1.1 This practice covers the digestion of soils and sediments for subsequent determination of acid-extractable concentrations of certain elements by such techniques as atomic absorption and atomic emission spectroscopy.

1.1.1 Concentrations of arsenic, cadmium, copper, lead, magnesium, manganese, nickel, and zinc can be extracted from the preceding materials. Other elements may be determined using this practice.

1.2 The analytical sample is arbitrarily defined as that which passes a 10-mesh (approximately 2-mm openings) screen and is prepared according to Practice D 3974.

1.3 Actual element quantitation can be accomplished by following the various test methods under other appropriate ASTM standards for element(s) of interest.

1.4 The detection limit and linear concentration range for each element is dependent on the atomic absorption or emission spectrophotometric technique employed and may be found in the manual accompanying the instrument used.

1.5 Before selecting a digestion technique, the user should consult the appropriate quantitation standard(s) for any special analytical considerations, and Practice D 3974 for any special preparatory considerations.

1.6 The extent of extraction of elements from soils and sediments by this method is dependent upon the physical and mineralogic characteristics of the prepared sample.

1.7 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.8 *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.* For specific hazard statements, see Section 8.

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. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

D 3974 Practices for Extraction of Trace Elements from Sediments³

2.2 *Code of Federal Regulations:*

CFR Title 21, Part 1030, and Title 47, Part 18⁴

3. Summary of Practice

3.1 The chemical portion of this practice involves acid digestion to dissociate the elements not interstitially bound in silicate lattices.

3.2 The sample is digested with nitric acid in a closed fluoropolymer vessel using microwave heating to an internal pressure of 100 psi (6.89×10^6 dynes/cm²).

3.3 This practice provides a sample suitable for analysis by atomic absorption or emission spectrophotometry.

4. Significance and Use

4.1 Partial extraction of soils and sediments can provide information on the availability of elements 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 nitric acid, reduces sample preparation or reaction times.

4.3 Little or no acids are lost to boiling or evaporation in the closed digestion vessel so additional portions of acid may not be required. Increased blank corrections from trace impurities in acid are minimized.

5. Interferences

5.1 No interferences to the digestion 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 elements using atomic absorption or emission spectroscopy.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

6. Apparatus

6.1 *Microwave Heating System*—A laboratory microwave heating system capable of delivering a minimum of 570 W of microwave energy. The system should be capable of 1 % power adjustments and 1 s time adjustment. The microwave cavity should be fluoropolymer coated and equipped with exhaust ventilation sufficient to provide ten chamber exchanges per minute. The cavity must have a 360° oscillating turntable to ensure even sample heating, and be capable of holding digestion vessels. Safety interlocks, to shut off magnetron power output, must be contained in the cavity door opening mechanism. The system must comply with Department of Health and Human Services Standards under 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 *Digestion Vessels*—A vessel of 100-mL capacity. The vessel must be transparent to microwave energy and be capable of withstanding a minimum internal pressure of 120 psi (8.27×10^6 dynes/cm²), and a temperature of 200°C. The vessel must contain a safety pressure relief valve, a rupture disc, pressure venting system, or be connected to an external safety relief valve that will prevent possible vessel rupture or ejection of the vessel cap.

6.3 *Pressure Control Vessel*—A vessel of 100 mL capacity, transparent to microwave energy, with a port for connection to a pressure control device and capable of withstanding a minimum internal pressure of 120 psi and temperature of 200°C.

6.4 *Pressure Control Device*—An externally or internally operated device to control the pressure within the digestion vessels. The controller must be capable of 1 psi (6.89×10^4 dynes/cm²) adjustments, controlling up to 100 psi and be equipped with an external pressure relief valve if a non-venting control vessel is used.

6.5 *Filtration Apparatus*—A gravity filter fitted with Whatman No. 41⁵ filter paper, or equivalent.

6.6 *Volumetric Flask*, 100-mL capacity.

7. Reagents

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 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.

⁵ Whatman No. 41 filter paper, available from Whatman Specialty Products, 6 Just Rd., Fairfield, NJ 07004, has been found suitable for this purpose.

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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III of Specification **D 1193**.

7.3 *Cleaning Solution*—Mix one volume of technical grade nitric acid (sp gr 1.42) with one volume water.

7.4 *Nitric Acid (1 + 1)*—Mix one volume nitric acid (sp gr 1.42) with one volume water.

8. Hazards

8.1 Operate and maintain the microwave system in accordance with the manufacturer's recommended safety precautions. Do not operate the microwave system in a fume hood where it is surrounded by acid fumes that can cause corrosion of the equipment. Vent acid fumes generated inside the cavity from the cavity to a fume hood. Place the digestion vessels in a fume hood to remove vapors released when a vessel is opened.

8.2 Perform the digestion in accordance with the manufacturer's recommended safety precautions.

9. Sampling

9.1 Collect a sediment sample using an appropriate sampling technique.

9.2 Prepare the sediment sample in accordance with Sections 10 and 12 of Practice **D 3974**.

10. Vessel Cleaning

10.1 The manufacturer's recommended cleaning procedure may be followed or the procedure in 10.2 through 10.4 may be used.

10.2 Soak the fluoropolymer vessel parts in cleaning solution at 60°C for 10 min.

10.3 Remove the vessel parts from the cleaning solution and thoroughly rinse the parts with tap water and then with reagent water.

10.4 Allow the vessel parts to air-dry or wipe dry using a clean, soft cloth.

11. Procedure

11.1 Determine the power output of the microwave using the procedure described in the annex to ensure that the unit meets the minimum power requirement.

11.2 Obtain a 1 g portion of the sample prepared in 9.2, weighed to the nearest 0.1 mg, and transfer into digestion vessels. Include an empty digestion vessel in each set as a method blank.

NOTE 1—The pressure control vessel must contain 1 g of sample material.

11.3 Add 20 mL of HNO₃(1 + 1) to each sample and blank digestion vessel.

11.4 Close each digestion vessel according to the manufacturer's recommended procedures.

11.5 Place the closed digestion and pressure control vessel into the instrument turntable and assemble following the manufacturer's suggested procedure.

NOTE 2—The pressure control vessel is connected to the pressure control device and may be assembled into the turntable differently than the