Designation: D5258 – 02 (Reapproved 2013)

Standard Practice for Acid-Extraction of Elements from Sediments Using Closed Vessel Microwave Heating[1]

This standard is issued under the fixed designation D5258; 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 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 D3974.

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 D3974 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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D3974 Practices for Extraction of Trace Elements from Sediments

2.2 *Code of Federal Regulations:*[3]

CFR Title 21, Part 1030, and Title 47, Part 18

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/cm^2).

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 leeching, 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.

[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] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://www.dodssp.daps.mil.

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.

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/cm^2), 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/cm^2) 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. 41[4] 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. [5] 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.

7.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III of Specification D1193.

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 D3974.

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.

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

[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 Recovery Data for Six Digestions of NIST River Sediment (SRM 1645) [A]

Element	Amount Present (µg/g)	Amount Recovered (µg/g)	Number of Digestions	Standard Deviation	Bias (µg/g)
Arsenic	66 (not cert.)	72	6	4	6
Cadmium	10.2 ± 1.5	12	6	5	0
Copper	109 ± 19	121	6	2	12
Lead	714 ± 28	726	6	8	12
Magnesium	7400 ± 200	7200	6	70	200
Manganese	785 ± 97	750	6	10	35
Nickel	45.8 ± 2.9	49	6	1	3
Zinc	1720 ± 170	1720	6	10	0

[A] See Footnote 7 for availability information.

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

11.3 Add 20 mL of HNO_3 (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 standard digestion vessels. Refer to the manufacturer's suggested procedure.

11.6 Set the pressure control device to control the digestion vessel pressure at 100 psi (6.89×10^6 dynes/cm^2).

11.7 Heat the vessels to obtain an internal pressure 100 psi (6.89×10^6 dynes/cm^2) and maintain for 30 min. Refer to the manufacturer's suggested procedure.

11.8 Allow the vessels to cool to room temperature, and then vent excess pressure from the vessels. Refer to the manufacturer's recommended venting procedure.

11.9 Remove the vessels from the turntable, place in a fume hood, and open the vessel.

11.10 Transfer the contents of the digestion vessel to a filtration device and filter. Wash the vessel and filter thoroughly with small portions of water. Alternatively, separate the solid and liquid phases by centrifugation.

11.11 Transfer the filtered solution and washes, or centrate, to a 100-mL volumetric flask and dilute to volume with water.

12. Precision and Bias

12.1 This practice was tested by digesting a single sample by one laboratory. Table 1 summarizes precision and bias of the trace element analyses conducted on six portions of NIST River Sediment (SRM 1645)[6] digested by this practice. All trace element concentrations were determined by flame or graphite furnace atomic absorption techniques.

13. Keywords

13.1 digestion; microwave; sediments; soils; vessel

[6] NIST Standard Reference Materials, Office of Standard Reference Materials, U.S. Department of Commerce, Gaithersburg, MD 20899.

ANNEX

(Mandatory Information)

A1. PROCEDURE TO DETERMINE DELIVERED POWER OF MICROWAVE OVEN AT 100 % INSTRUMENT POWER

A1.1 Remove the turntable, drive lug, and all vessels from the instrument cavity.

A1.2 Adjust the instrument cavity exhaust to minimum airflow.

A1.3 Program the instrument for 4 min time and 100 % power.

A1.4 Transfer 2000± 2 mL of room temperature (19 to 25°C) water into a 2-L polypropylene beaker.

A1.5 Measure and record the initial water temperature, T_i, to the nearest 0.1°C.

A1.6 Place the beaker in the right front corner of the instrument cavity.

A1.7 Heat the water for the programmed time.

A1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature, T_f, to the nearest 0.1°C.

A1.9 Calculate the delivered power as follows:

$$P = (T_f - T_i) \times 35$$

where:

P = delivered power, watts,
T_f = final water temperature, °C,
T_i = initial water temperature,° C
and:

$$35 = \frac{4.2 \times 1.0 \times M}{t}$$

where:

4.2 = conversion factor for thermochemical calories to watts,
1.0 = heat capacity of water, calories/g per °C,
M = mass of water, g, and
t = heating time, s.