Designation: ~~D5258 – 02 (Reapproved 2013)~~ D5258 – 21

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.~~mathematical conversions to SI units that are provided for information only and are not considered standard.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 8.

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[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:*[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~~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.

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~~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.

[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.