



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

This standard is issued under the fixed designation D 5258; 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 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.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

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.

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.

Current edition approved July 10, 2002. Published July 2002. Originally published as D 5258 – 92. Last previous edition D 5258 – 92 (1996).

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.