



Designation: E2941 – 13

Standard Practice for Nitric Acid-Extraction of Elements from Ores and Related Metallurgical Materials by Closed Vessel Microwave Heating¹

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1. Scope

1.1 This practice covers the digestion of ores and related metallurgical materials, such as mine soil, waste rock and tailings, for subsequent determination of nitric acid-extractable contents of certain elements by such solution analytical techniques as atomic absorption, atomic emission (see Method D1976), and inductively coupled plasma mass spectroscopy (see Method D5673).

1.1.1 Contents of aluminum, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chromium, cobalt, copper, gallium, iron, lead, lithium, magnesium, manganese, mercury, molybdenum, nickel, phosphorus, potassium, scandium, selenium, silver, sodium, strontium, thallium, tin, titanium, vanadium and zinc can be extracted from ores and related metallurgical materials for determination by analytical methods for elements in solution. Other elements may be determined from extracts produced using this practice.

1.1.2 Actual element quantification can be accomplished by following the various test methods under other appropriate ASTM standards for element(s) of interest in solution.

1.1.3 The detection limit and linear content range for each element is dependent on the atomic absorption or emission spectrometric technique employed and may be found in the manual accompanying the instrument used or ASTM standard method for analysis of the solutions. Take into account the 100 fold dilution due to digesting a 1-g sample to final volume of 100 mL.

1.1.4 The extent of extraction of elements from ores and related metallurgical materials by this method is dependent upon the physical and mineralogical characteristics of the prepared sample.

1.2 The values stated SI units are to be regarded as the standard. No other units of measurements are included in this standard.

1.3 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 9.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy

D5258 Practice for Acid-Extraction of Elements from Sediments Using Closed Vessel Microwave Heating

D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

2.2 Federal Standard:³

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

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E135.

4. Summary of Practice

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

¹ This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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 U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

4.2 The sample is digested with HNO_3 in a closed fluoropolymer vessel using microwave heating to an internal pressure of 6.89×10^5 Pa.

4.3 This practice provides a sample suitable for analysis by atomic absorption, atomic emission or inductively coupled plasma mass spectrometry.

5. Significance and Use

5.1 These test methods are primarily intended to test materials for compliance with compositional specifications and for monitoring. Partial extraction of ores and related metallurgical materials can provide information on the availability of elements to leaching, water quality changes, or other site conditions.

5.2 Rapid heating, in combination with temperatures in excess of the atmospheric boiling point of HNO_3 , reduces sample preparation or reaction times.

5.3 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.

5.4 It is assumed that the users of this practice will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices such as those described in Guide E882 must be followed.

6. Interferences

6.1 No interferences to the digestion of ores and related metallurgical materials using microwave heating have been observed.

6.2 Precautions should be exercised to avoid those interferences normally associated with the final determination of elements using atomic absorption, atomic emission, or inductively coupled plasma mass spectrometry.

6.3 The HNO_3 matrix may not be suitable for stabilizing solutions containing silver and antimony, an alternate digestion method using a HCl matrix may be required to determine some elements.

7. Apparatus

7.1 *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.

7.1.1 *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 8.27×10^5 Pa, 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.

7.1.2 *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 8.27×10^5 Pa and temperature of 200°C .

7.1.3 *Pressure Control Device*—An externally or internally operated device to control the pressure within the digestion vessels. The controller must be capable of 6.89×10^3 Pa adjustments, controlling up to 8.27×10^5 Pa and be equipped with an external pressure relief valve if a non-venting control vessel is used.

7.2 Other commercially available laboratory microwave heating systems may be used so long as they can be shown to provide comparable safety and performance. Follow manufacturers instruction for use.

8. Reagents

8.1 *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.⁴ 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or Type II of Specification D1193. Type III or Type IV may be used if they effect no measurable change in the blank or sample.

9. Hazards

9.1 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.

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

⁴ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.