



# Standard Practices for Extraction of Elements from Ores and Related Metallurgical Materials by Acid Digestion<sup>1</sup>

This standard is issued under the fixed designation E2941; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These practices cover the digestion of ores and related metallurgical materials, such as mine soil, waste rock and tailings, for subsequent determination of acid-extractable contents of certain elements by such solution analytical techniques as atomic absorption spectrometry (AAS), atomic emission spectrometry (ICP-AES), (see Test Method [D1976](#)), and inductively coupled plasma mass spectrometry (ICP-MS) (see Test 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 in digested solutions 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, mass spectrometry 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 dilution factor in content calculations due to digestion and dilution of solid samples.

1.1.4 The extent of extraction of elements from ores and related metallurgical materials by these practices is dependent upon the physical and mineralogical characteristics of the prepared sample and the digestion practice used.

1.2 The digestion practices appear in the following order:

Nitric Acid Microwave Digestion	Sections
Four-Acid Total Digestion	7 to 14
	15 to 21.12

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

1.4 *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 11.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

- [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:*<sup>4</sup>

- [CFR Title 21, Part 1030, and Title 47, Part 18](#)

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in these practices, refer to Terminology [E135](#).

<sup>1</sup> 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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<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> 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. Significance and Use

4.1 These practices 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.

4.2 It is assumed that the users of these practices 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 shall be followed.

#### 5. Reagents

5.1 *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.<sup>5</sup> 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.

5.2 *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.

#### 6. Sampling and Sample Preparation

6.1 *Materials Safety*—Samples shall be prepared, stored, and disposed of in accordance with the materials and safety guidelines in Practices E50.

6.2 *Prepared Sample*—Dry a representative portion of the gross sample at 80 °C to constant mass in order to minimize sulfide mineral oxidation. Pulverize or grind the laboratory sample until at least 95 % passes a 150- $\mu$ m sieve. Fine grinding to 95 % passing a 53- $\mu$ m sieve is recommended for procedures using a single acid digestion.

### NITRIC ACID MICROWAVE DIGESTION

#### 7. Summary of Practice

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

7.2 The sample is digested with HNO<sub>3</sub> in a closed fluoropolymer vessel using microwave heating to an internal pressure of 6.89  $\times$  10<sup>5</sup> Pa.

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

<sup>5</sup> *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 the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

#### 8. Significance and Use

8.1 Rapid heating, in combination with temperatures in excess of the atmospheric boiling point of HNO<sub>3</sub>, reduces sample preparation or reaction times.

8.2 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.

8.3 HNO<sub>3</sub> digestion of ores and related metallurgical materials are most useful for rapid, low-cost digestions, where metals locked in the silica or other insoluble matrix components are not important for the results of the analysis.

#### 9. Interferences

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

9.2 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.

9.3 The HNO<sub>3</sub> matrix may not be suitable for stabilizing solutions containing silver and antimony; an alternate digestion method using a HCl or HF matrix may be required to determine some elements (i.e. silicon).

#### 10. Apparatus

10.1 *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 shall 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, shall be contained in the cavity door-opening mechanism. The system shall 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.

10.1.1 *Digestion Vessels*—A vessel of 100-mL capacity. The vessel shall be transparent to microwave energy and have an operating capability to withstand an internal pressure of at least 8.27  $\times$  10<sup>5</sup> Pa, and a temperature of 200 °C. The vessel shall 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.

10.1.2 *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 an internal pressure of at least 8.27  $\times$  10<sup>5</sup> Pa and temperature of 200 °C.