INTERNATIONAL

Designation: ~~E2941 – 14~~ E2941 – 21

Standard Practices for Extraction of Elements from Ores and Related Metallurgical Materials by Acid Digestion[1]

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 (ε) 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), inductively coupled plasma atomic emission spectrometry ~~(ICP-AES),~~(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~~ Consider 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:

	Sections
Nitric Acid Microwave Digestion	7 to 14
~~Four-Acid Total Digestion~~	~~15 to 21.12~~
Four-Acid Total Digestion	15 to 21

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

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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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see ~~Section~~Sections 11 and 20.*

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
D1976 Test Method for Elements in Water by Inductively-Coupled 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 these practices, refer to Terminology E135.

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.[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.

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.

[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] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, ~~DC.~~ DC, www.chemistry.org. 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.~~MD, www.usp.org.

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-µm sieve. Fine grinding to 95 % passing a 53-µm 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~~HNO_3 ~~acid~~ digestion to dissociate the elements not interstitially bound in silicate lattices.

7.2 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.

7.3 This practice provides a sample suitable for analysis by ~~atomic absorption, atomic emission, or inductively coupled plasma mass spectrometry.~~AAS, ICP-AES, or ICP-MS.

8. Significance and Use

8.1 Rapid heating, in combination with temperatures in excess of the atmospheric boiling point of HNO_3, 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_3 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_3 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.~~ for example, 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 ~~shallbe~~ shall be transparent to microwave energy and have an operating capability to withstand an internal pressure of at least 8.27×10^5 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 8.27×10^5 Pa and temperature of 200 °C.

10.1.3 *Pressure Control Device*—An externally or internally operated device to control the pressure within the digestion vessels. The controller shall 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.

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

11. Hazards

11.1 For hazards to be observed in the use of reagents and apparatus in this practice, refer to Practices E50.

11.2 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.

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

11.4 Use of other domestic and commercial microwave systems not designed for nitricHNO_3 acid digestion of samples may not be suitable for use with this practice.

12. Preparation of Apparatus

12.1 The manufacturer's recommended cleaning procedure may be followed or the following procedure may be used.

12.2 Soak the fluoropolymer vessel parts in HNO_3 (1 + 1) cleaning solution at 60 °C ± 5 °C for 10 min.

12.3 Remove the vessel parts from the cleaning solution and thoroughly rinse the parts with water.

12.4 Allow the vessel parts to air-dry or wipe dry using a clean, soft cloth.

13. Procedure

13.1 Determine the power output of the microwave using the procedure described in the annex to ensure that the unit meets the minimum power requirement.

13.2 Obtain a 1 g portion of the sample prepared in 6.2, weighed to the nearest 0.1 mg, and transfer into digestion vessels. Include an empty digestion vessel in each set as a method blank. The pressure control vessel shall contain 1 g of sample material.

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

13.3 Add 20 mL of HNO_3 (1 + 1) to each sample and blank digestion vessel.

13.4 Close each digestion vessel according to the manufacturer's recommended procedures.

13.5 Place the closed digestion and pressure control vessel into the instrument turntable and assemble following the manufacturer's suggested procedure. 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.

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.

13.6 Set the pressure control device to control the digestion vessel pressure at 6.89×10^5 Pa.

13.7 Heat the vessels to obtain an internal pressure 6.89×10^5 Pa and maintain for 30 min. Refer to the manufacturer's suggested procedure.

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

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

13.10 Filter[5] the contents of the digestion vessel. Wash the vessel and filter thoroughly with small portions of water, without exceeding 100 mL solution volume. Alternatively, separate the solid and liquid phases by centrifugation.

13.11 Transfer the filtered solution and washes, or concentrate, to a 100-mL volumetric flask and dilute to volume with water. The 100-mL volumetric flasks may receive the filtrate directly from the funnel.

~~NOTE 3—The 100-mL volumetric flasks may receive the filtrate directly from the funnel.~~

13.12 Apply the 100-fold dilution to the analysis of the solution to calculate the solid material content.

14. Precision and Bias

14.1 This practice was tested by digesting a single sample by one laboratory during the standardization of Practice D5258. 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 contents were determined by flame or graphite furnace atomic absorption techniques.

FOUR-ACID TOTAL DIGESTION

15. Summary of Practice

15.1 The sample is weighed into a fluoropolymer beaker, four acids are added, and samples are digested to dryness on a hotplate in a fume hood. The residues are re-dissolved in HCl transferred to tubes and quantitatively diluted.

15.2 This practice provides a sample suitable for analysis by AAS,ICP-AES, or ICPMS.

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 6 for availability information.

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

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