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

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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:

	Sections
Nitric Acid Microwave Digestion	7 to 14
Four-Acid Total Digestion	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:*[3]

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:*[4]

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.

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[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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[3] 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.

[4] 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.[5] 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-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 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.

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. 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 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.

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[5] *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.

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 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 nitric 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.

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.

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[6] 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.

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.

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.

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

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)[7] 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.

16. Significance and Use

16.1 This digestion method is applicable to solid, pulverized samples for determination of total metals by AAS or ICP-AES. Analytes that fall below the established detection limit of the ICP-AES can be easily determined by ICP-MS following a pre-dilution of the solution.

16.2 Alternative digestion practices are more appropriate for determination of silicon, arsenic, antimony, and selenium, due to volatile losses during acid fuming.

17. Interferences

17.1 No interferences to the digestion of ores and related metallurgical materials using four-acid total digestion have been observed.

17.2 Silica is lost during the volatilization of the acid fumes and cannot be determined by this practice; arsenic, antimony, and selenium may also be lost during acid fuming.

18. Apparatus

18.1 *Fluoropolymer Beakers,* 250-ml capacity.

18.2 *Fluoropolymer Watch Glass,* for 250-mL capacity beaker.

18.3 *Hot Plate,* capable of digesting samples in fluoropolymer beakers to dryness.

19. Reagents and Materials

19.1 *d-Tartaric Acid.*

19.2 *d-Tartaric Acid Solution (10 % w/v)*—In a 100-mL volumetric flask, dissolve 10.0 g d-tartaric acid crystals in ~30 mL water. Bring to volume and mix thoroughly.

19.3 *Polypropylene Tubes,* 10-mL and 50-mL capacity, with volume markings.

20. Hazards

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

20.2 This method involves the use of concentrated acids and strong oxidizers at elevated temperatures. Concentrated HNO_3 and HCl are highly corrosive and both evolve irritating fumes.

20.3 $HClO_4$ is a strong oxidizer that poses an explosive property on contact with organic substances. Only fume hoods certified for the use of perchloric acid should be used.

20.4 HF is a known contact hazard and is highly toxic.

20.5 All operations should be performed in a laboratory fume hood. A laboratory coat, safety glasses, and protective gloves should be worn at all times. As with all tests, the primary responsibility for safety lies with the analyst. Consult the appropriate MSDS and the chemical hygiene plan, if available, for further details.

21. Procedure

21.1 Weigh 0.5 g + 0.01 g of sample into a 250-mL fluoropolymer beaker.

21.2 In a fume hood, dispense the acids in the following order: 10 mL water, 10 mL HNO_3, 10 mL $HClO_4$, 5 mL HF

21.3 Swirl to mix acids and place the beakers on the hot plate and cover with fluoropolymer watch glasses for 30 min.

21.4 Rinse watch glasses with water into the beaker and remove watch glasses. Continue to digest to dryness. Heavy white fumes will evolve from the samples during the process.

NOTE 4—Keep a close eye on the samples as they reduce in volume. If the beakers are left unsupervised after drying they will melt.

21.5 Remove the beakers from the hotplate and allow to cool.

21.6 Add 10 mL water and then 10 mL HCl. **CAUTION: Water must be dispensed before the HCl or an explosion may occur or acid may (violently) spatter due to an exothermic reaction with water.**

21.7 Return the beakers to the hotplate and reheat uncovered for about 5 min. This step is meant only to re-dissolve the residue.

NOTE 5—Allowing the samples to reheat beyond the allotted time will cause the volatilization of HCl(g) and will result in loss of Ag through precipitation.

21.8 While still warm, transfer sample into a labeled 50 mL polyethylene tube. Cool to ambient temperature and add 0.5 mL d-Tartaric Acid Solution (19.2) and bring to a final volume of 50 mL with water. Cap tightly and shake to mix.

NOTE 6—d-Tartaric Acid Solution (19.2) is for stabilization of Sb and Bi.

21.9 Allow samples to settle overnight, or, if analysis is needed immediately, centrifuge an aliquot.

21.10 Pipet 1.00 mL and dilute to 10 mL with (1 + 4) HCl.

21.11 Shake diluted tubes to mix well.

21.12 Apply the 1000-fold dilution to the analysis of the solution

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[7] NIST Standard Reference Materials, Office of Standard Reference Materials, U.S. Department of Commerce, Gaithersburg, MD 20899.

22. Keywords

22.1 atomic absorption spectrometry; digestion; inductively coupled plasma atomic emission spectrometry; inductively coupled plasma mass spectrometry; microwave; mine soils; ores and related metallurgical materials; tailings; vessel; waste rock

ANNEX

(Mandatory Information)

A1. PROCEDURE TO DETERMINE DELIVERED POWER OF MICROWAVE OVEN AT 100 % INSTRUMENT POWER

A1.1 Remove the turntable, drive lug, and all vessels from the instrument cavity.

A1.2 Adjust the instrument cavity exhaust to minimum airflow.

A1.3 Program the instrument for 4 min time and 100 % power.

A1.4 Transfer 2000 mL ± 2 mL of room temperature (19 °C to 25 °C) water into a 2-L polypropylene beaker.

A1.5 Measure and record the initial water temperature, T_i, to the nearest 0.1 °C.

A1.6 Place the beaker in the right front corner of the instrument cavity.

A1.7 Heat the water for the programmed time.

A1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature, T_f, to the nearest 0.1 °C.

A1.9 Calculate the delivered power as follows:

$$P = (T_f - T_i) \times 35 \tag{A1.1}$$

where:

P = delivered power, watts,
T_f = final water temperature, °C, and
T_i = initial water temperature, °C.

and

$$35 = \frac{4.2 \times 1.0 \times M}{t} \tag{A1.2}$$

where:

4.2 = conversion factor for thermochemical calories to watts,
1.0 = heat capacity of water, calories/g per °C,
M = mass of water, g, and
t = heating time, s.

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