Designation: D1971 – 16 (Reapproved 2021)ε1

Standard Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry[1]

This standard is issued under the fixed designation D1971; 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.

ε1 NOTE—The WTO caveat was editorially added in November 2021.

1. Scope

1.1 Most atomic absorption and plasma emission spectroscopy, and plasma-mass spectrometric test methods require that the metals of interest be dissolved in a liquid phase before being introduced into the spectrophotometer. These practices describe digestion or dissolution procedures whereby analyte metals associated with the solid fraction of a sample can be brought into solution for subsequent analysis. The following practices are included:

	Sections
Practice A—Digestion with Mineral Acids and Elevated Pressure	8 through 13
Practice B—Digestion with Mineral Acids and Heating at Atmospheric Pressure	14 through 19
Practice C—In-Bottle Digestion with Mineral Acids	20 through 25

1.2 These practices have been demonstrated to be applicable to a wide variety of sample types and sample matrices, and in many cases, will give complete dissolution of the analyte metals of interest. They are by no means the only digestion procedures available.

1.3 The user of these practices should be cautioned that these practices may not completely dissolve all portions of a sample's solid phase and may not give complete recovery of the desired analyte metals. In these cases, other digestion techniques are available that will effect complete dissolution of a sample. It is the user's responsibility to ensure the validity of these practices for use on their particular sample matrix, for their metals of interest.

1.4 This practice assumes that the criteria established in Guide D3856 can be met.

1.5 These digestion procedures have been selected for their wide application, low cost, and ease of use.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are mathematical conversion to inch-pound units that are provided for information only and are not considered standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

1.8 *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]

D511 Test Methods for Calcium and Magnesium In Water
D857 Test Method for Aluminum in Water
D858 Test Methods for Manganese in Water
D1068 Test Methods for Iron in Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1687 Test Methods for Chromium in Water
D1688 Test Methods for Copper in Water
D1691 Test Methods for Zinc in Water
D1886 Test Methods for Nickel in Water
D1976 Test Method for Elements in Water by Inductively-Coupled Plasma Atomic Emission Spectroscopy
D2972 Test Methods for Arsenic in Water
D3082 Test Method for Boron in Water

[1] These practices are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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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.

D3370 Practices for Sampling Water from Flowing Process Streams
D3372 Test Method for Molybdenum in Water
D3373 Test Method for Vanadium in Water
D3557 Test Methods for Cadmium in Water
D3558 Test Methods for Cobalt in Water
D3559 Test Methods for Lead in Water
D3645 Test Methods for Beryllium in Water
D3697 Test Method for Antimony in Water
D3859 Test Methods for Selenium in Water
D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
D3866 Test Methods for Silver in Water
D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D3920 Test Method for Strontium in Water
D4190 Test Method for Elements in Water by Direct-Current Plasma Atomic Emission Spectroscopy
D4191 Test Method for Sodium in Water by Atomic Absorption Spectrophotometry
D4192 Test Method for Potassium in Water by Atomic Absorption Spectrophotometry
D4309 Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Metals in Water
D4382 Test Method for Barium in Water, Atomic Absorption Spectrophotometry, Graphite Furnace
D4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry
D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry

2.2 *EPA Method:*[3]

EPA-600/4-79-020 Methods for Chemical Analysis of Water and Wastes, Revised March 1983
EPA-600/R-94/111 Methods for the Determination of Metals in Environmental Samples—Supplement 1[3]

2.3 *USGS Method:*[4]

USGS Open File Report 96–225 Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—In-Bottle Acid Digestion of Whole Water Samples

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *digestion, n*—treating a sample with the use of heat or elevated pressures, or both, usually in the presence of chemical additives, to bring analytes of interest into solution or to remove interfering matrix components, or both.

3.2.2 *total recoverable, n*—a descriptive term relating to the metal forms recovered in the acid-digestion procedures resulting in a metal analyte measurable by atomic absorption spectrophotometry, plasma emission spectroscopy or plasma mass spectrometry after applying the digestion procedure in either Practice A, Practice B, or Practice C.

3.2.2.1 *Discussion*—The choice of Practice A, B, or C shall be noted in reporting resultant data.

4. Significance and Use

4.1 The determination of metals in water often requires the measurement of total (suspended and dissolved) metals as well as soluble (dissolved) metals. In such cases, consistent and dependable digestion procedures must be used so that data derived for the total metals content is reliable.

4.2 The practices given are applicable to a wide variety of sample types for the purpose of preparing a sample for metals analyses by atomic absorption spectrophotometry or plasma emission spectroscopy (see Test Method D1976, Practice D3919, Practice D4691, and Test Method D4190) or plasma-mass spectrometry (see Test Method D5673) and have been shown to give good recovery in the following matrices: industrial effluents; waste water treatment plant influents, sludges, dewatered sludges, and effluents; river and lake waters; and plant and animal tissues. Elements which have shown good recovery include: copper, nickel, lead, zinc, cadmium, iron, manganese, magnesium, and calcium.

4.2.1 Good recovery for the indicated sample types and metals may not be achieved at all times due to each sample's unique characteristics. Users must always validate the practice for their particular samples.

4.3 The analytical results achieved after applying these practices cannot necessarily be deemed as a measure of bioavailable or environmentally available elements.

4.4 These three practices may not give the same recovery when applied to the same sample, nor will they necessarily give the same results as achieved using other digestion techniques. An alternate digestion technique is Practice D4309.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used throughout. Acids shall have a low-metal content or should be doubly distilled and checked for purity. 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.[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 Specification D1193, Type I. Other reagent water types may be used, provided it is first ascertained that the water is of

[3] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

[4] Available from U.S. Geological Survey (USGS) National Center, 12201 Sunrise Valley Dr., Reston, VA 20192, https://www.usgs.gov.

[5] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.