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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 Spectrometry¹

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.

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 for information only.

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

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](#) ~~D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits~~

D1193 [Specification for Reagent Water](#)

D1687 [Test Methods for Chromium in Water](#)

D1688 [Test Methods for Copper 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.

*A Summary of Changes section appears at the end of this standard.

- D1691 Test Methods for Zinc in Water
- D1886 Test Methods for Nickel in Water
- D1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy
- D3370 Practices for Sampling Water from Closed Conduits
- D3372 Test Method for Molybdenum 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
- D3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
- D3866 Test Methods for Silver in Water
- D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
- 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
- D4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry
- D5673 Test Method for Elements in Water by Inductively Coupled PlasmaMass Spectrometry

2.2 U.S. EPA Method:

EPA-600/4-79-020 Methods for Chemical Analysis of Water and Wastes³

EPA-600/R-94/111 Methods for the Determination of Metals in Environmental Samples—Supplement 1³

2.3 U.S. Geological Survey Method:

U.S. Geological Survey 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

Quality Laboratory—In-Bottle Acid Digestion of Whole Water Samples^{4,4}

3. Terminology

3.1 *Definitions*: For definitions of terms used in these practices, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *digestion*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*total recoverable, n—the amount of 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. The choice of Practice A, B, or C shall be noted in reporting resultant data.

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 Methods D1976, D3919, D4691, and 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

³ Revised March 1983. Published by the United States Environmental Protection Agency. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁴ Available from USGS National Center, 12201 Sunrise Valley Dr., Reston, VA 20192, USA.

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 sufficiently high purity to permit its use without lessening the bias and precision of the determination.

6. Hazards

6.1 These practices involve the heating of solutions of mineral acids. Appropriate precautions shall be taken to protect the analyst from these acids and heated containers. Heated samples and acids may splatter or boil unexpectedly.

7. Sampling

7.1 As with all chemical assay procedures, the user of this practice shall ensure that all sample aliquot used are adequately representative of the environmental situation being monitored.

7.2 Appropriate sampling and subsampling techniques for particular environmental samples can be found in other references.

~~7.3 Collect the sample in accordance with Specification D1192 and Practices~~

7.3 Collect the sample in accordance with Practice D3370.

PRACTICE A—DIGESTION WITH MINERAL ACIDS AND ELEVATED PRESSURE

8. Scope

8.1 This practice presents a digestion technique that has broad application and can be performed inexpensively with minimal labor, equipment, and space. In addition, this practice allows for many samples to be processed quickly and simultaneously under the same conditions.

9. Summary of Practice

9.1 Samples are placed in loosely capped, heat-, and acid-resistant containers with selected reagents and subjected to 121°C and 15 psig (103 kPa gage) for 30 min. After removing any particulate matter remaining, the digestate is ready for analysis by atomic absorption spectrophotometry, plasma emission spectroscopy, or plasma-mass spectrometry.

9.2 The practice may be found to be more applicable to a particular sample or analytical scheme after appropriate modifications of reagent addition, temperature, pressure, digestion time, or container selection. Any such modifications to this practice must be validated by the user.

10. Apparatus

10.1 *Digestion Containers*—50 mL disposable polypropylene centrifuge tubes and 125 mL polypropylene reagent bottles with screw caps have been used successfully. Any container that is not attacked by the digestion conditions, is sufficiently free of the analyte(s) of interest, and can be loosely capped, may be used.

10.2 *Digestion Container Rack*—Any rack that will fit inside the autoclave, will hold the digestion containers securely, and is not attacked by the conditions in the autoclave, may be used.

10.3 *Autoclave*—Any autoclave or similar apparatus with a pressure chamber large enough to hold the desired number of samples and capable of achieving and holding 121°C and 15 psig (103 kPa gage) for 30 min., may be employed. An autoclave with automatic cycling is desirable. As the digesting samples release acidic fumes, the portions of the autoclave coming in contact with these fumes should be constructed of acid resistant materials.

NOTE 1—Prolonged use of an autoclave with a stainless steel interior for this practice may result in discoloration of the autoclave walls. This discoloration has not been shown to cause any problems with autoclave operation. A commercially available autoclave with a stainless steel interior has been in daily use for this practice, as well as for routine sterilization purposes, for ten years without any degradation of the autoclave or its performance.

11. Interferences

11.1 The interferences of this practice relate to the inability of the described procedure to quantitatively dissolve the analyte metals of interest in certain situations. These interferences can be either physical or chemical.

11.2 *Physical Interferences*—In some samples, the metals of interest are bound or occluded in a matrix that is impervious to dissolution by the acids. This is most frequently encountered in geological and boiler water samples.

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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.