Designation: D4382 – 12

Standard Test Method for Barium in Water, Atomic Absorption Spectrophotometry, Graphite Furnace[1]

This standard is issued under the fixed designation D4382; 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 This test method covers the determination of dissolved and total recoverable barium in most waters and wastewaters.

1.2 This test method was evaluated in the range from 33.5 to 132 µg/L of barium. The range can be increased or decreased by varying the volume of sample injected or the instrumental settings. High concentrations may be diluted but preferably should be analyzed by direct aspiration atomic absorption spectrophotometry.

1.3 This test method has been used successfully with waste treatment plant effluent water, lake water, filtered tap water, and well water. It is the responsibility of the analyst to determine the suitability of the test method for other matrices.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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
D1886 Test Methods for Nickel in Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D2972 Test Methods for Arsenic 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
D3859 Test Methods for Selenium in Water
D3866 Test Methods for Silver in Water
D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total recoverable barium, n*—an arbitrary analytical term relating to the recoverable forms of barium that are determinable by the digestion method which is included in this test method.

4. Summary of Test Method

4.1 Barium is determined by an atomic absorption spectrophotometer used in conjunction with a graphite furnace. A sample is placed in a graphite tube, evaporated to dryness, charred (pyrolyzed or ashed), and atomized. The absorption signal produced during atomization may be recorded and compared with values obtained from standards that have been carried through the same process. This facilitates interpolation of the level of barium in the solution being analyzed. Since the graphite furnace uses the sample much more efficiently than flame atomization, the detection of low concentrations in small sample volumes is possible.

NOTE 1—The same graphite furnace procedure may be applicable to

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is 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

determination of arsenic (see Test Methods D2972), cadmium (see Test Methods D3557), chromium (see Test Methods D1687), cobalt (see Test Methods D3558), copper (see Test Methods D1688), iron (see Test Methods D1068), lead (see Test Methods D3559), manganese (see Test Methods D858), nickel (see Test Methods D1886), selenium (see Test Methods D3859), silver (see Test Methods D3866), and vanadium (see Test Method D3373).

4.2 Dissolved barium is determined on a sample filtered through a 0.45-µm membrane filter. The definition of dissolved barium is arbitrary since very fine crystals of barium sulfate may pass through the membrane filter.

4.3 Total recoverable barium is determined following acid digestion and filtration. Because chlorides interfere with furnace procedures for some metals, the use of hydrochloric acid in any digestion or solubilization step is to be avoided. If suspended material is not present, this digestion and filtration may be omitted. The holding time for the samples may be calculated in accordance with Practice D4841.

5. Significance and Use

5.1 Barium ranks about sixth in order of abundance in nature; however, it is normally found in only trace quantities in drinking water. Consumption, inhalation, or absorption of 500 to 600 mg is considered fatal to human beings. Lower levels may result in disorders of the heart, blood vessels, and nerves. The drinking water standards set the maximum contaminant level for barium as 2 mg/L.[3] Lower levels may result in disorders of the heart, blood vessels, and nerves. The drinking water standards set the maximum contaminant level for barium as 2 mg/L.

6. Interferences

6.1 For a complete discussion on general interferences with furnace procedures, refer to Practice D3919.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer,* for use at 553.6 nm with background correction. A general guide for flame atomic absorption applications is given in Practice D4691.

NOTE 2—A wavelength other than 553.6 nm may be used if it has been determined to be suitable. At high concentration, greater linearity may be obtained by using a less sensitive wavelength.

NOTE 3—The manufacturer's instructions should be followed for all instrumental parameters.

7.2 *Barium Light Source*—Barium hollow-cathode lamp. A single-element lamp is preferred. Multielement lamps containing calcium are not recommended.

7.3 *Graphite Furnace,* capable of reaching temperatures sufficient to atomize the element of interest.

7.4 *Graphite Tubes,* compatible with furnace device. To eliminate the formation of carbides, pyrolytically coated graphite tubes are recommended.

7.5 *Data Storage and Reduction Devices*—Computer and microprocessor controlled devices, or a strip chart recorder, shall be utilized for data collection, storage, reduction, and problem recognition (drift, incomplete atomization, changes in sensitivity, etc.).

7.6 *Automatic Sampling* accessory should be used, if available.

8. Reagents and Materials

8.1 *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.

8.2 *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 adversely affecting the bias and precision of the test method. Type II water was specified at the time of round-robin testing of this test method.

8.3 *Barium Solution, Stock* (1.0 mL = 1000 µg barium)—Dissolve 1.779 g of barium chloride ($BaCl_2 \cdot 2H_2O$) in 50 mL of concentrated hydrochloric acid (HCl) (sp gr 1.19) and about 700 mL of water. Dilute to 1 L with water. A purchased barium stock solution of appropriate known purity is also acceptable.

8.4 *Barium Solution, Intermediate* (1.0 mL = 10 µg barium)—Dilute 10.0 mL of barium solution, stock (8.3) and 1 mL of HNO_3 (sp gr 1.42) to 1 L with water.

8.5 *Barium Solution, Standard* (1.0 mL = 0.10 µg barium)—Dilute 10.0 mL of barium intermediate solution (8.4) and 1 mL of HNO_3 (sp gr 1.42) to 1 L with water. This standard is used to prepare working standards at the time of the analysis.

8.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

NOTE 4—If the reagent blank concentration is greater than the method detection limit, distill the HNO_3 or use a spectrograde acid.

8.7 *Argon,* standard, welders grade, commercially available. Nitrogen and hydrogen may also be used, if recommended by the instrument manufacturer.

9. Standardization

9.1 Initially, set the instrument in accordance with the manufacturer's specifications. Follow the general instructions as provided in Practice D3919.

[3] *Standards Method for the Examination of Water and Wastewater,* 15th Edition, American Public Health Assn., 1015 15th St., NW, Washington, DC 20005.

[4] *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 *Annual 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.