



## Standard Test Methods for Beryllium in Water<sup>1</sup>

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### 1. Scope

1.1 These test methods cover the determination of dissolved and total recoverable beryllium in most waters and wastewaters:

	Concentration Range	Sections
Test Method A—Atomic Absorption, Direct	10 to 500 $\mu\text{g/L}$	7 to 16
Test Method B—Atomic Absorption, Graphite Furnace	10 to 50 $\mu\text{g/L}$	17 to 25

1.2 The analyst should direct attention to the precision and bias statements for each test method. It is the user's responsibility to ensure the validity of these test methods for waters of untested matrices.

1.3 *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 12, 23.4, and Note 2, Note 6, Note 7, and Note 9.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 858 Test Methods for Manganese in Water<sup>2</sup>
- D 1068 Test Methods for Iron in Water<sup>2</sup>
- D 1129 Terminology Relating to Water<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 1687 Test Methods for Chromium in Water<sup>2</sup>
- D 1688 Test Methods for Copper in Water<sup>2</sup>
- D 1691 Test Methods for Zinc in Water<sup>2</sup>
- D 1886 Test Methods for Nickel in Water<sup>2</sup>
- D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup>
- D 3557 Test Methods for Cadmium in Water<sup>2</sup>
- D 3558 Test Methods for Cobalt in Water<sup>2</sup>
- D 3559 Test Methods for Lead in Water<sup>2</sup>
- D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry<sup>2</sup>

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents<sup>2</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total recoverable beryllium*—total recoverable beryllium relates only to the recoverable forms of beryllium by these test methods.

### 4. Significance and Use

4.1 These test methods are significant because the concentration of beryllium in water must be measured accurately in order to evaluate potential health and environmental effects.

### 5. Purity of Reagents

5.1 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.<sup>3</sup> Other grades may be used, provided it is first ascertained that the reagent is sufficiently high in purity to permit its use without lessening the accuracy of the determination.

5.2 Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, 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.

### 6. Sampling

6.1 Collect the samples in accordance with Practices D 3370. The holding time for samples may be calculated in accordance with Practice D 4841.

6.2 Preserve samples with  $\text{HNO}_3$  (sp gr 1.42), adding about 2 mL/L, to a pH of 2 or less immediately at the time of

<sup>3</sup> *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

collection. If only dissolved beryllium is to be determined, filter the sample, before acidification, through a 0.45- $\mu\text{m}$  membrane filter.

## TEST METHOD A—ATOMIC ABSORPTION, DIRECT

### 7. Scope

7.1 This test method is applicable in the range from 10 to 500  $\mu\text{g/L}$  of beryllium. The range may be extended upward by dilution of the sample.

7.2 The precision and bias data were obtained on reagent water, tap water, salt water, river water, lake water, spring water, and untreated wastewater. The information on precision and bias may not apply to other waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

### 8. Summary of Test Method

8.1 Beryllium is determined by atomic absorption spectrophotometry. Dissolved beryllium is determined by aspirating a filtered sample directly with no pretreatment. Total recoverable beryllium in the sample is determined in a portion of the filtrate obtained after a hydrochloric-nitric acid digestion of the sample. The same digestion procedure is used to determine total recoverable cadmium (Test Methods D 3557), chromium (Test Methods D 1687), cobalt (Test Methods D 3558), copper (Test Methods D 1688), iron (Test Methods D 1068), lead (Test Methods D 3559), manganese (Test Methods D 858), nickel (Test Methods D 1886), and zinc (Test Methods D 1691).

### 9. Interferences

9.1 Aluminum at concentrations greater than 500  $\mu\text{g/L}$  has been reported to depress the beryllium absorbance.

9.2 Sodium and silicon at concentrations in excess of 1000  $\text{mg/L}$  have been reported to severely depress the beryllium absorbance.

9.3 Beryllium is slightly ionized in the nitrous oxide-acetylene flame. This ionization is suppressed by adding calcium chloride to give a final concentration of 900  $\text{mg/L}$  calcium in all standard and sample solutions.

### 10. Apparatus

10.1 *Atomic Absorption Spectrophotometer*, for use at 234.9 nm.

NOTE 1—The manufacturer's instructions should be followed for all instrument parameters.

10.2 *Beryllium Hollow Cathode Lamp*.

10.3 *Pressure Regulators*—The supplies of oxidants and fuel shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable regulators.

### 11. Reagents and Materials

11.1 *Beryllium Solution, Stock* (1.00 mL = 1000  $\mu\text{g}$  Be)—Dissolve 1.000 g of beryllium metal in a minimum volume of HCl (1 + 1) and dilute to 1 L.

NOTE 2—**Warning:** Beryllium is toxic and the solution should be prepared in a well-ventilated hood.

NOTE 3—Certified beryllium stock solutions are commercially available through chemical supply vendors.

11.2 *Beryllium Solution, Intermediate* (1.00 mL = 100  $\mu\text{g}$  Be)—Dilute 10.0 mL of the beryllium stock solution to 100.0 mL with nitric acid ( $\text{HNO}_3$ , 1 + 499).

11.3 *Beryllium Solution, Standard* (1.00 mL = 1.00  $\mu\text{g}$  Be)—Dilute 5.00 mL of the beryllium intermediate solution to 500.0 mL with nitric acid ( $\text{HNO}_3$ , 1 + 499).

11.4 *Calcium Solution* (10 g/L)—Dissolve 25 g of calcium carbonate in a minimum volume of HCl (1 + 1) and dilute to 1 L with water.

11.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

NOTE 4—If a high reagent blank is obtained, distill the HCl or use spectrograde acid. **Caution**—When HCl is distilled, an azeotropic mixture is obtained (approximately 6 N HCl). Therefore, whenever concentrated HCl is specified in the preparation of a reagent or in the procedure, use double the amount if distilled acid is used.

11.6 *Hydrochloric Acid* (1 + 1)—Mix 1 volume of HCl (sp gr 1.19) with 1 volume of water.

11.7 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid ( $\text{HNO}_3$ ).

NOTE 5—If a high reagent blank is obtained, distill the  $\text{HNO}_3$  or use spectrograde acid.

11.8 *Nitric Acid* (1 + 499)—Add 1 volume of  $\text{HNO}_3$  (sp gr 1.42) to 499 volumes of water.

11.9 *Oxidant:*

11.9.1 *Air*, which has been passed through a suitable filter to remove oil, water, and other foreign substances, is the oxidant used prior to switching to nitrous oxide.

11.9.2 *Nitrous Oxide* is the required oxidant.

11.10 *Fuel:*

11.10.1 *Acetylene*—Standard commercially available acetylene is the required fuel. Acetone, always present in acetylene cylinders, can affect analytical results. The cylinder should be replaced at a gage pressure of 75 psi (517 kPa).

NOTE 6—**Warning:** "Prepurified" grade acetylene containing a special proprietary solvent other than acetone should not be used with poly(vinyl chloride) tubing as weakening of the walls can cause a potentially hazardous situation.

### 12. Hazards

12.1 Due to the high toxicity of beryllium, all sample preparation and digestion steps should be carried out in a well-ventilated hood. Also, the atomic absorption unit should be vented as recommended by the manufacturer.

### 13. Standardization

13.1 Prepare a blank and at least four standard solutions to bracket the expected beryllium concentration range of the samples to be analyzed by diluting the beryllium standard solution (11.3) with  $\text{HNO}_3$ (1 + 499). Prepare the standards (100 mL) each time the test is to be performed.

13.2 For total recoverable beryllium, add 0.5 mL of  $\text{HNO}_3$ (sp gr 1.42) and proceed as directed in 14.2-14.6. For dissolved beryllium, proceed with 13.3.

13.3 Add 1.0 mL of calcium solution to a 10.0-mL aliquot of each standard and blank solution. Mix thoroughly.