



Designation: D3645 – 15 (Reapproved 2023)

Standard Test Methods for Beryllium in Water¹

This standard is issued under the fixed designation D3645; 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 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 $\mu\text{g/L}$ to 500 $\mu\text{g/L}$	7 to 17
Test Method B—Atomic Absorption, Graphite Furnace	10 $\mu\text{g/L}$ to 50 $\mu\text{g/L}$	18 to 26

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 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 12 and 24.4.

1.5 *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

D858 Test Methods for Manganese in Water

¹ These test methods 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.

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
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Flowing Process Streams
D3557 Test Methods for Cadmium in Water
D3558 Test Methods for Cobalt in Water
D3559 Test Methods for Lead in Water
D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry
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 these test methods, refer to Terminology D1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total recoverable beryllium, n*—a descriptive term relating to the beryllium forms recovered in the acid-digestion procedure specified in 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.³ 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 **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.

6. Sampling

6.1 Collect the samples in accordance with Practices **D3370**. The holding time for samples may be calculated in accordance with Practice **D4841**.

6.2 Preserve samples with HNO_3 (sp gr 1.42), adding about 2 mL/L, to a pH of 2 or less immediately at the time of collection. If only dissolved beryllium is to be determined, filter the sample, before acidification, through a 0.45 μm membrane filter.

NOTE 1—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. This could reduce hazards of working with acids in the field when appropriate.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

7. Scope

7.1 This test method is applicable in the range from 10 $\mu\text{g/L}$ 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 **D3557**), chromium (Test Methods **D1687**), cobalt (Test Methods **D3558**), copper (Test Methods **D1688**), iron (Test Methods **D1068**), lead (Test Methods **D3559**), manganese (Test Methods **D858**), nickel (Test Methods **D1886**), and zinc (Test Methods **D1691**).

³ 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.

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 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 mg/L calcium in all standard and sample solutions.

10. Apparatus

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

NOTE 2—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 μg Be)—Dissolve 1.000 g of beryllium metal in a minimum volume of HCl (1 + 1) and dilute to 1 L. (Beryllium is toxic and the solution should be prepared in a well-ventilated hood.) A purchased metal stock solution of appropriate known purity is also acceptable.

11.2 *Beryllium Solution, Intermediate* (1.00 mL = 100 μg Be)—Dilute 10.0 mL of the beryllium stock solution to 100.0 mL with nitric acid (HNO_3 , 1 + 499).

11.3 *Beryllium Solution, Standard* (1.00 mL = 1.00 μg Be)—Dilute 5.00 mL of the beryllium intermediate solution to 500.0 mL with nitric acid (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 *Filter Paper*—Purchase suitable filter paper. Typically the filter papers have a pore size of 0.45 μm membrane. Material such as fine-textured, acid-washed, ashless paper, or glass fiber paper are acceptable. The user must first ascertain that the filter paper is of sufficient purity to use without adversely affecting the bias and precision of the test method.

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

NOTE 3—If a high reagent blank is obtained, distill the HCl or use spectrograde acid. (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.)

11.7 *Hydrochloric Acid* (1 + 1)—Mix 1 volume of HCl (sp gr 1.19) with 1 volume of water. Always add acid to water.

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