Designation: D3082 – 15

Standard Test Method for Boron in Water[1]

This standard is issued under the fixed designation D3082; 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 boron in water and wastewaters by the curcumin colorimetric-extraction method[2] in concentrations between 0.1 and 1.0 mg/L. The range can be extended by dilution of the sample.

1.2 Only dissolved boron is determined. This test method requires that the water sample be filtered through a 0.45-µm membrane filter before analysis.

1.3 This test method is a colorimetric method that is very sensitive to low concentrations of boron in water and requires a relatively small sample volume for analysis.

1.4 Precision and bias were obtained on natural and wastewaters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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

1.6 *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:*[3]

D1066 Practice for Sampling Steam
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
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
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

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

4. Summary of Test Method

4.1 When a water sample containing soluble boron is acidified with hydrochloric acid and evaporated to dryness in the presence of curcumin, a red-colored complex called rosocyanine is formed. This colored product is taken up in isopropyl alcohol and is read spectrophotometrically.

5. Significance and Use

5.1 Because boron can be both essential and deleterious to plant growth, and because ingestion of large amounts can affect the central nervous system in humans, a method is required to determine its concentration in potable, natural, and wastewaters. This test method provides a means of determining the boron concentration of these waters. The holding time for the samples may be calculated in accordance with Practice D4841.

5.2 Boric acid is used for chemical shim control of neutron flux in a nuclear reactor. This test method serves to determine if the boron concentration is within acceptable limits.

6. Interferences

6.1 Nitrate concentrations above 20 mg/L begin to interfere. Hardness levels about 100 mg/L as $CaCO_3$ give high results because of the turbidity caused by the insolubility of the hardness salts in isopropyl alcohol. The turbidity can be eliminated by filtering the final solution through a 0.45-µm membrane filter before reading on the spectrophotometer.

[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] This test method is similar to, but not identical with that appearing in *Standard Methods for Examination of Water and Wastewater*, 13th Ed., American Public Health Association, Washington, DC, pp 69–72.

[3] 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**

6.2 Organic color may be present in the sample that could affect absorbance readings on the spectrophotometer. If an interfering organic color is present in the sample, the following procedure has been found useful in reducing this interference for some matrices. Pipet an appropriate sample aliquot into a platinum dish (Note 1). Make alkaline to litmus with NaOH solution (20 g/L) and add 3 drops in excess. Evaporate to dryness on a steam or hot-water bath. If desired, organic material may be destroyed by ignition from 500 to 550°C before proceeding. Allow the platinum dish to cool and acidify with 5 mL of HCl (1 + 19). Triturate with a rubber policeman to dissolve the residue, pour the contents into a calibrated centrifuge tube, wash the platinum dish with 3 or 4 mL of water, and add to the centrifuge tube. Dilute to the 10-mL mark. Centrifuge to obtain a clear solution. Perform the same steps on a reagent blank.

NOTE 1—Other types of evaporating dishes may be used but must be checked. Porcelain or ceramic-type dishes may contain boron-fluxing agents.

7. Apparatus

7.1 All laboratory ware used in the performance of this test method must either be plastic or boron-free.

7.2 *Hot-Water Bath,* with temperature control at 55 ± 2°C.

7.3 *Spectrophotometer,* suitable for use in the range of 540 nm. The photometric practices prescribed in this test method shall conform to Practice E60. Spectrophotometers shall conform to Practice E275. Measure absorbance using a 50-mm cell.

7.4 *Evaporating Dishes,* 100 to 150 mL capacity.

7.5 *Thermometer*—An ASTM Gravity Thermometer having a range from –20 to +102°C, as specified, and conforming to the requirements for Thermometer 12C as prescribed in Specification E2251.

8. Reagents and Materials

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, 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, II, or III water. Type I is preferred and more commonly used. Type II water was specified at the time of round robin testing of this test method.

NOTE 2—The user must ensure the type of reagent water chosen is sufficiently free of interferences. The water should be analyzed using the test method.

8.3 *Boron Solution, Stock* (1.00 mL = 1.00 mg B)—Dry about 10 g of boric acid (H_3BO_3) crystals in a desiccator containing a silica gel desiccant for 24 h (Note 2). Dissolve 5.719 g of the dry H_3BO_3 in water and dilute to 1 L. Store the solution in a plastic bottle or boron-free container. Alternatively, certified boron stock solutions of appropriate known purity are commercially available through chemical supply vendors and may be used.

NOTE 3—If boric acid is heated, it gradually loses water, changing first to metaboric acid (HBO_2) and finally dehydrating completely to the anhydrous oxide (B_2O_3). It is important therefore that oven drying not be used as a method of drying boric acid.

8.4 *Boron Solution, Standard* (1.00 mL = 0.010 mg B)—Quantitatively dilute 10.0 mL of the stock boron solution to 1 L with water. Store in a plastic bottle or boron-free container.

8.5 *Curcumin Solution*—Dissolve 40 mg of finely ground curcumin [5] and 5 g of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) in 80 mL of isopropyl alcohol. Add 4.0 mL of hydrochloric acid (HCl, sp gr 1.19) and make up to 100 mL with isopropyl alcohol.

NOTE 4—There are other colorimetric methods available for boron testing which use other indicators (the Carmine Method).

8.6 *Hydrochloric Acid* (1 + 19)—Add 1 volume of hydrochloric acid (sp gr 1.19) to 19 volumes of water.

8.7 *Isopropyl Alcohol.*

8.8 *Sodium Hydroxide Solution* (20 g/L)—Dissolve 2 g of NaOH in water and dilute to 100 mL.

8.9 *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.

9. Sampling

9.1 Collect the samples in accordance with Practice D1066 or Practices D3370.

9.2 Filter the sample through a 0.45-µm membrane filter as soon as possible after sampling.

9.3 Samples should be collected and stored in polyethylene bottles or alkali-resistant, boron-free glass. No other preservation is required.

10. Calibration and Standardization

10.1 Prepare a series of standard boron solutions to cover the range from 0 to 1.0 mg/L. Make up standards by diluting suitable volumes of the boron standard solution (1.00 mL = 0.010 mg B) to 100 mL.

[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.

[5] The sole source of supply of curcumin known to the committee at this time is Eastman No. 1179. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.