# Boron, colorimetric, curcumin

Parameters and Codes:

Boron, dissolved, I-1112-85 (μg/L as B): 01020 Boron, total recoverable, I-3112-85 (μg/L as B): 01022 Boron, suspended recoverable, I-7112-85 (μg/L as B): 01021

# 1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100  $\mu$ g/L of boron. The optimum range for the method on undiluted or unconcentrated samples is from 100 to 1,000  $\mu$ g/L.

1.2 Suspended recoverable boron is calculated by subtracting dissolved boron from total recoverable boron.

1.3 Total recoverable boron in water-suspended sediment needs to undergo a preliminary digestion-solubilization by method I-3485 before being determined.

## 2. Summary of method

2.1 When a sample of water containing boron is acidified and evaporated in the presence of curcumin, a red-colored product, called rosocyanine, is formed. The rosocyanine is extracted into a suitable solvent and the red color, which has a maximum absorbance at 540 nm, is measured spectrometrically.

2.2 The method is identical to that found in "Standard Methods for the Examination of Water and Wastewater" (American Public Health Association and others, 1980).

#### 3. Interferences

3.1 Nitrate-nitrogen concentrations greater than 20 mg/L interfere.

3.2 Significantly high results are possible when the total of calcium and magnesium hardness exceeds 100 mg/L as calcium carbonate. Moderate hardness levels also can cause a considerable percentage error in the low boron range. The interference arises from the insolubility of the hardness salts in 95-percent ethanol and consequent turbidity in the final solution. Filter the final solution or pass the original sample through a column of strongly acidic cationexchange resin in the hydrogen form to remove the interfering cations. The latter procedure enables application of the method to waters and effluents of high hardness or solids content.

# 4. Apparatus

4.1 Evaporating dish, 100- to 150-mL capacity, of Vycor glass, platinum, or other suitable material.

4.2 Ion-exchange column, 50-cm long by 1.3-cm diameter.

4.3 Spectrometer, for use at 540 nm, and cells with a minimun light-path length of 1 cm.

4.4 Refer to manufacturer's manual to optimize instrument.

4.5 Water bath,  $55\pm2$  °C.

## 5. Reagents

5.1 Boron standard solution I, 1.00 mL=100  $\mu$ g B: Dissolve 0.5720 g high-purity H<sub>3</sub>BO<sub>3</sub>, dried over desiccant for 24 h, in demineralized water and dilute to 1000 mL. Store in plastic bottle.

5.2 Boron standard solution II, 1.00 L=1.00  $\mu$ g B: Dilute 10.0 mL boron standard solution I to 1,000 mL with demineralized water. Store in plastic bottle.

5.3 Cation-exchange resin: Load the column with a strongly acidic cation-exchange resin. Backwash the column with demineralized water to remove entrained air bubbles. Henceforth, make certain the resin remains covered with liquid at all times. Pass 50 mL 2*M* HCl through the column at a rate of 0.2 mL/min of acid per milliliter of resin in column and then wash it free of acid with demineralized water. The frequency of regeneration depends on the mineral content of the samples.

5.4 Curcumin reagent: Dissolve 40 mg finely ground curcumin (Eastman No. 1179 or equivalent) and 5.0 g oxalic acid in 80 mL 95-percent ethanol. Add 4.2 mL concentrated HCl (sp gr 1.19) and dilute to 100 mL with ethanol. The reagent is stable for several days if stored in a refrigerator.

5.5 Ethanol, 95-percent.

5.6 Hydrochloric acid, 2M: Mix 166 mL concentrated HCl (sp gr 1.19) with demineralized water and dilute to 1 L.

## 6. Procedure

6.1 *Precautions*: Exercise close control of variables, such as volumes and concentrations of samples, standards, and, reagents, as well as time and temperature of drying. Use evaporating dishes identical in shape, size, and composition to insure equal evaporation time. Increasing the time of evaporation results in intensification of the resulting color.

6.2 For samples having high hardness (100 mg/L CaCO<sub>3</sub> or more), proceed as follows (NOTE 1): Pipet 25 mL of sample solution or of a smaller sample of known high boron content diluted to 25 mL onto the resin column. Adjust the rate of flow through the column to about 2 drops per second and collect the effluent in a 50-mL volumetric flask. Wash the column with small portions of demineralized water until the flask is full to the mark and mix.

NOTE 1. For samples containing less than 100 mg/L hardness as  $CaCO_3$ , start with paragraph 6.3.

6.3 Pipet 1.00 mL of sample solution containing less than 1.0  $\mu$ g B into an evaporating dish (NOTE 2).

NOTE 2. If the sample contains more than 1,000  $\mu$ g/L B, make an appropriate dilution with demineralized water, so that a 1.00-mL portion contains approx 0.50  $\mu$ g B.

6.4 Prepare in evaporating dishes a blank and sufficient standards (1.0  $\mu$ g B max) and adjust the volume of each to exactly 1.0 mL with demineralized water.

6.5 Add 4.0 mL curcumin reagent to each and swirl each dish gently to mix contents.

6.6 Float the dishes on a water bath set at  $55\pm2$  °C and let them remain for 80 min, a time

usually sufficient for complete drying and removal of HCl. Keep drying time constant for standards and samples.

6.7 After the dishes cool to room temperature, add 10.0 mL 95-percent ethanol to each dish, stirring gently with a polyethylene rod to ensure complete dissolution of the red-colored product.

6.8 Wash the contents of each dish into a 25-mL volumetric flask using 95-percent ethanol and adjust to volume with ethanol. Mix by repeated inversion (NOTE 3).

NOTE 3. If the final sample solution is turbid, filter through No. 30 Whatman filter paper or equivalent.

6.9 Determine the absorbance of samples and standards against the blank. Complete all absorbance readings within 1 h of drying the samples.

## 7. Calculations

7.1 Determine the micrograms boron in the sample solution from a plot of absorbances of standards.

7.2 Determine the dissolved or total-recoverable boron concentration in micrograms per liter as follows:

B (
$$\mu g/L$$
) =  $\frac{1,000 \times \mu g \text{ B in sample.}}{\text{mL sample}}$ 

7.3 To determine micrograms per liter of suspended recoverable boron, subtract dissolved-boron concentration from total-recoverable-boron concentration.

#### 8. Report

Report boron, dissolved (01020), total-recoverable (01022), and suspended-recoverable (01021), concentrations as follows: less than 1,000  $\mu$ g/L, nearest 100  $\mu$ g/L; 1,000  $\mu$ g/L and above, two significant figures.

#### 9. Precision

9.1 The standard deviation for dissolved boron within the range of 65 to 643  $\mu$ g/L for 16 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 63.4  $\mu$ g/L ranged from 54.2 to 76.3  $\mu$ g/L. 9.2 Precision for dissolved boron for four of the 16 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
4	65	37
5	98	60
4	320	20
6	643	10

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable boron will be greater than that reported for dissolved boron.

## Reference

American Public Health Association and others, 1980, Standard methods for the examination of water and wastewater (15th ed.): Washington, D.C., p. 257.