

Boron, atomic emission spectrometric, d-c plasma

Parameter and Code:

Boron, dissolved, I-1114-85 ($\mu\text{g/L}$ as B): 01020

1. Application

This method may be used to analyze finished water, natural water, and industrial water containing from 10 to 1000 $\mu\text{g/L}$ of boron. Samples containing more than 1000 $\mu\text{g/L}$ boron and/or with specific conductances greater than 10,000 $\mu\text{S/cm}$ need to be diluted.

2. Summary of method

Boron is determined by a direct-reading emission spectrometer that utilizes a d-c argon plasma as an excitation source (Johnson and others, 1979a, b, 1980). A mixture of lithium chloride, sulfuric acid, and glycerin is added to samples and standards to provide a common background matrix and to compensate for viscosity changes. The liquid mixture is then converted by a ceramic nebulizer into a fine aerosol and introduced into the plasma via a plastic spray chamber and Pyrex injection tube. Boron is determined on the basis of the average of two replicate exposures, each of which is performed on a 10 second integrated intensity. Calibration is performed by standardization with a high- standard solution and a blank.

3. Interferences

Stray-light effects in a high-resolution, single-element, d-c argon plasma emission spectrometer are found to be negligible.

4. Apparatus

4.1 *Spectrometer*, Spectrometrics, Spectrospan IV with dc argon plasma or equivalent, with Echelle optics, printer, autosampler, and peristaltic pump.

4.2 Refer to manufacturer's manual to optimize instrument for the following:

Plasma viewing position ----- 1 (fig. 1)
Gas ----- Argon
Sleeve pressure ----- 50 psi
Nebulizer pressure ----- 25 psi
Entrance slit ----- 25 X 300 μm
Exit slit ----- 50 X 300 μm
Voltage ----- 1000 V
Wavelength ----- 249.773 nm
Signal amplification ----- 40 to 60 percent full-scale (1000 $\mu\text{g/L}$)

5. Reagents

5.1 *Boron standard solution I*, 1.00 mL = 100 $\mu\text{g B}$: Dissolve 0.5720 g high-purity H_3BO_3 , 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 mL = 10.0 $\mu\text{g B}$: Dilute 100.0 mL boron standard solution I to 1000 mL with demineralized water. Store in plastic bottle.

5.3 *Boron working standard*, 1.00 mL=1.00 $\mu\text{g B}$: Dilute 100.0 mL boron standard solution II to 1000 mL with demineralized water. Store in plastic bottle.

5.4 *Glycerin*, USP.

5.5 *Lithium chloride*, LiCl, reagent-grade.

5.6 *Matrix modifier*: Dissolve 367 g LiCl in 1000 mL demineralized water. Transfer to a Teflon beaker and add, with stirring, 10.0 mL concentrated H_2SO_4 . Heat the solution to 75 to 80°C on a hotplate (asbestos padded) and slowly add 25 mL methyl alcohol. Stir rapidly for one hour to volatilize of excess methyl alcohol and any trimethyl borate that forms. Repeat the

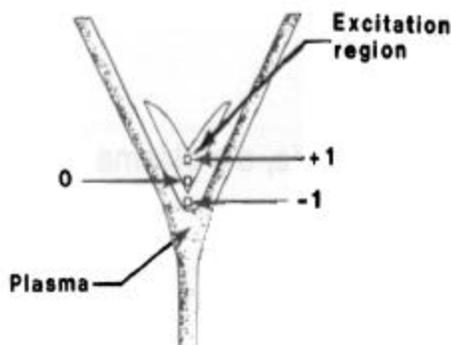


Figure 1.-Plasma position on entrance slit for boron

process two more times. Allow the solution to cool, transfer to a 4-L polyethylene container, and add with stirring 2000 mL glycerin. In a Teflon beaker slowly add, with stirring, 400 mL concentrated H₂SO₄ to 400 mL demineralized water. With stirring and cooling, add 50 mL methyl alcohol. The heat generated should be sufficient to volatilize excess methyl alcohol and any trimethyl borate. When the acid has reached room temperature, add the acid slowly, with stirring (in a hood), to the glycerin-LiCl mixture. Dilute to 4,000 mL with demineralized water.

5.7 Methyl alcohol, reagent-grade.

5.8 Sulfuric acid, concentrated (sp gr 1.84), Ultrex or equivalent.

6. Procedure

6.1 Pipet 10.0 mL sample into a disposable plastic test tube.

6.2 Pipet 100 mL demineralized-water blank and working standard into plastic bottles.

6.3 Add 2.0 mL matrix modifier to the sample and 20.0 mL to the blank and working standard.

6.4 Place plastic caps on the tube and bottles and mix well.

6.5 Refer to manufacturer's manual for computer-operating procedure. Use the prepared blank and working standard for instrument calibration and all subsequent recalibrations.

6.6 Refer to manufacturer's manual for auto-sampler-operating procedures. Pour samples in autosampler tray, positioning a blank and working standard after every five samples for recalibration. Begin analysis (NOTE 1).

NOTE 1. Because of thermal instability inherent with the high-resolution spectrometer, repeak the analytical line if the boron standard drifts more than 3 percent.

7. Calculations

The computer system is designed so that the blank and the 1000 µg/L of boron working standard are used to establish a two-point calibration curve. The system will convert instrument-intensity readings to analytical concentrations. The printer display includes the blank and working-standard instrument intensity readings, blank and standard concentrations, sample instrument intensity readings, sample concentrations, average of sample concentrations, and standard deviation.

8. Report

Report boron, dissolved (01020), concentrations as follows: less than 100 µg/L, nearest 10 µg/L; 100 µg/L and above, two significant figures.

9. Precision

Precision based on 12 determinations by a single operator expressed in terms of standard deviation and percent relative standard deviation is as follows:

Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
30.8	1.8	5.8
40.8	1.8	4.4
215	5.0	2.3
425	1.3	0.3

References

- Johnson, G.W., Taylor, H.E., and Skogerboe, R.K., 1979a, Determination of trace elements in natural waters by the D.C. Argon-plasma, multielement atomic emission spectrometer (DCP-MAES) technique: *Spectrochimica Acta*, v. 34B, p. 197-212.
- _____, 1979b, Evaluation of spectral interferences associated with a direct current plasma-multielement atomic emission spectrometer (DCP-MAES) system: *Applied Spectroscopy*, v. 33, p. 451-456.
- _____, 1980, Characterization of an interelement enhancement effect in a dc plasma atomic emission spectrometry system: *Applied Spectroscopy*, v. 34, p. 19-24.