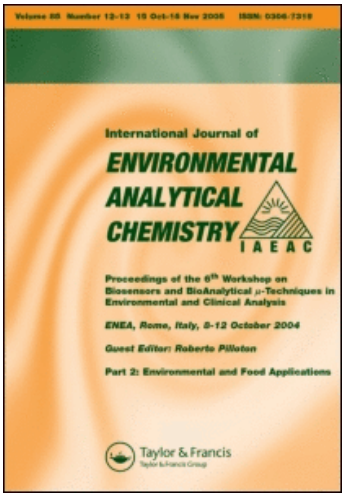

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OSCILLOPOLAROGRAPHIC DETERMINATION OF TRACE AMOUNT OF BORON IN SOIL, WATER AND PLANT SAMPLES

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Since boron is a necessary trace nutrient element in the physiological development of growing plants, it is important to be able to determine accurately the total amount of boron, soluble boron, and residual boron in soil samples, the boron in environmental water, and the boron in plant samples.

Boron with Beryllon III can produce a stable ionic complex in buffer solution (pH 4–4.5).[1] The complex yields a well-defined high-sensitivity polarographic peak at a potential of about −0.46 V (vs SCE). Under optimum conditions, the wave height is proportional to the concentration of boron in the range from 0.004 to 0.4 μg/ml. The method has been used to determine trace amounts of boron in environmental water samples, total boron, soluble boron and residual boron in soil samples, and trace amounts of boron in plant samples.

EXPERIMENTAL

Apparatus

A JP-2 oscillopolarograph (Chengdu Instrumental Factory, China) was used. For derivative polarography with the JP-2 oscillopolarograph, the conditions were: drop time 7 sec, scan rate 250 mV/sec, scan from −0.25 to 0.75 V, mercury head 50 cm, and mercury flow rate 2.0 mg/sec. The three-electrode system comprised a dropping mercury electrode (DME), platinum counter-electrode, and saturated calomel electrode (SCE) as reference. All potentials to the SCE. The electrolytic cell was a 10-ml beaker.

Reagents

Unless otherwise mentioned, all reagents used were of analytical reagent grade.

Buffer solution, ammonium acetate–acetic acid, pH 3.9.
Beryllon III solution, 0.04%.
Triethanolamine, 20%, v/v in water.
EDTA, 10% solution.
Lithium hydroxide (solid reagent).

Standard boron solution A stock solution containing 0.5 mg of boron per ml is

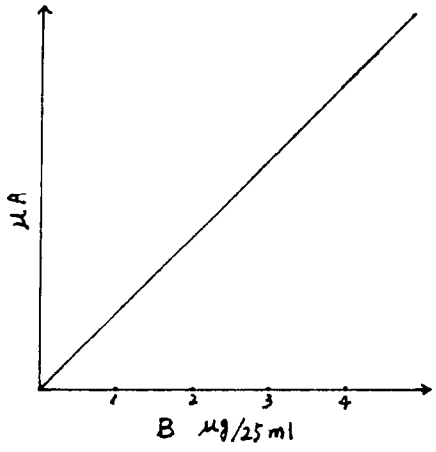


Figure 1 Calibration graph.

prepared by dissolving 0.286 g boric acid in water. The solution is finally diluted to give a working solution in water, containing 1 μg of B per ml.

Procedure

Calibration graph Transfer 0.1–10 μg of B into 25 ml volumetric flasks. Add 3 ml of ammonium acetate–acetic acid solution (pH 3.9). Add 2.5 ml of a 0.04% Beryllon III solution. Make up the contents to the mark with water, mix and heat in 90 °C water bath for 8 min. After cooling, transfer some solution to the polarographic cell. Record the derivative linear-sweep polarogram from −0.25 to 0.75 V (vs SCE). Measure the peak heights of the polarogram at −0.46 V (vs SCE) in the usual way. Usually the oxygen dissolved in the solutions need not be removed. Draw the calibration graph (Figure 1). The wave shape is shown in Figure 2.

Determination of Boron in Environmental Water Samples

Transfer 5–10 ml of sample water into a 25 ml volumetric flask. Add 3 ml of ammonium acetate–acetic acid solution (pH 3.9). Add 1 ml of EDTA solution (10%). Add 1 ml of TEA solution (20%), and mix. Add 2.5 ml of Beryllon III solution (0.04%). Make up to the mark with water, and mix. Determine boron as described above. At the same time draw the calibration graph (the additional amounts of EDTA and TEA are equal in quantity to those used in this determination).

Determination of the Soluble Boron in Soil Samples

To a 5 g soil sample in a polytetrafluoroethylene (PTFE) crucible, add two drops

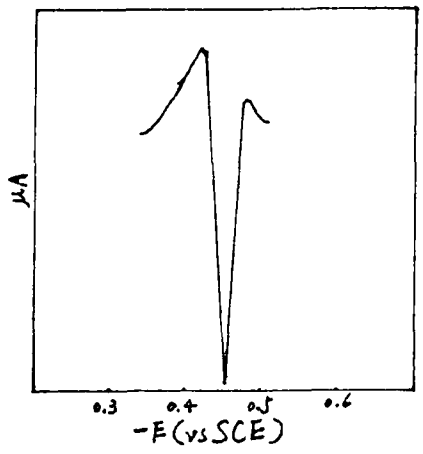


Figure 2 The derivative linear-sweep polarogram of boron.

of calcium chloride solution (1 mol/L). Add 10 ml of boiling water with new aluminium cooking-pot (avoid pollution of B).[3] Maintain the temperature of the water for 30 min. Transfer 2–3 ml of the clear solution into a 25 ml volumetric flask. Add 3 ml of ammonium acetate–acetic acid solution (pH 3.9). Add 2 ml of EDTA solution (10%). Add 3 ml of TEA solution (20%), and mix. Add 3 ml of Beryllon III solution (0.04%). Make up the contents to the mark with water, and mix. Determine boron as described above. At the same time draw the calibration graph.

Determination of Total Amount of Boron in Soil Samples

Fuse 0.5 g sample of soil in a nickel crucible with 3 g of lithium hydroxide at 650 °C, and leach the cooled melt with 6 M hydrochloric acid in a PTFE beaker. Add barium carbonate as paste to neutralize excess acid, and boil the solution. After cooling, transfer the solution and precipitate in the beaker into a 100 ml volumetric flask. Make up the contents to the mark with water, and mix. Transfer 5–10 ml of the clear solution into a 25 ml volumetric flask. Determine boron as in determination of the soluble boron in soil sample described above.

Calculation of residue boron of soil sample:

$$\text{Residue boron} = \text{Total boron} - \text{Soluble boron.}$$

Determination of Boron in Plant Sample

Take a 0.5 g plant sample in a porcelain crucible (in the case of the seed sample, a little amount of saturated solution of calcium hydroxide is added, this is done for stabilizing B).[2] Slowly raise the temperature to 200 °C, and keep for 30 min. Then

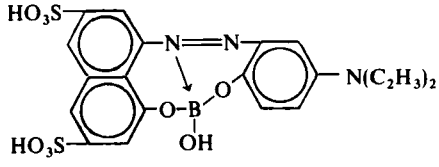
slowly raise the temperature to 500 °C, and keep for 40 min. After cooling, dissolve the ash in 15 ml of hydrochloric acid (0.1 M). Add 0.5 g barium carbonate (solid), neutralizing the excess hydrochloric acid, and boil. After cooling, transfer into a 25 ml volumetric flask. Make up the contents to the mark with water, and mix.

Transfer 5 ml of this supernatant into a 25 ml volumetric flask. Add 3 ml of ammonium acetate–acetic acid solution (pH 3.9). Determine boron as in determination of the boron in environmental water described above.

RESULTS AND DISCUSSION

Constitution of Boron–Beryllon III Complex

The composition of boron–Beryllon III complex has been shown to be 1:1 boron–Beryllon III by the isomolar continuous-variations and slop-ratio methods. A possible structure is described below:



Nature of the Boron Wave

No wave was observed for a solution of boron at the peak potential of −0.46 V (vs. SCE) in the absence of Beryllon III.

The relationship between the height of the mercury column and the peak height, the relative temperature coefficient of the peak height for boron, the electrocapillary curves, the log-log plot of current vs. time, all of these indicate that, the boron wave is due to adsorption of the boron complex with Beryllon III on the surface of the mercury drop.

The Effect of Common Ions

More than 40 anions and cations were examined for possible interference in the determination of boron by LSP. The results proved that most of the cations have little effect. Only aluminium and iron interfere seriously. The effect of these elements is removed with EDTA and TEA. Anions do not interfere with the determination of boron.

Table 1 Analytical results (B μg/g)

Soil sample	*Present method*	*Certified value*	*Plant sample*	*Present method*	*Certified value*
1	50.4	50	01	25.0	27
2	39.0	36	02	13.5	13
3	23.4	23	03	44.5	45
4	52.6	53	04	57.0	56

Samples 1, 2, 3 and 4 are standard samples of geological ministry of China, the results are total amounts of B.

Samples 01, 02, 03 and 04 are from Lab. of Hebei Agri. Univ.

Results of Analysis

The results shown in Table 1 are in good agreement with the certified values.

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