Application Bulletin

Metrohm

Of interest for:
Laboratories for water analysis and the determination of trace elements in soils and plants

No. 44/1 e

Colorimetric determination of boron

Summary

The Bulletin describes a colorimetric method for the determination of boron with the aid of curcumin, when boron is present in very small quantities.

Apparatus

- 2.662.0010 Photometer with light guide

Reagents

- **Curcumin solution**
  dissolve 0.125 g curcumin in 100 mL glacial acetic acid with slight warming, and filter into a dark-glass bottle.
- **Caustic soda**
  10% NaOH solution.
- **Sulphuric/acetic acid mixture**
  take 50 mL 96% pure glacial acetic acid and add slowly, with cooling, 50 mL conc. H_2SO_4.
- **Extraction solution**
  make a mixture of 5 parts methylethyl ketone and 2 parts chloroform, and dissolve 10 mg AR grade phenol in 100 mL of this mixture. This solution should be freshly prepared every day.
- **Standard boric acid solution**
  10^{-2} mol/L H_3BO_3 solution should be kept in stock, and the necessary 10^{-5} or 10^{-6} mol/L solution made from it by dilution shortly before the experiment is begun.

Method

- According to the amount of boron present, take a sample of 5 to 20 mL, add 1 mL of the caustic soda solution, and dry over a water-bath in a platinum or quartz evaporating dish. Then add 3 mL of the curcumin reagent, and heat the dish in an oven at 60 °C for 10 min, shaking it occasionally to dissolve the residue. After cooling to room temperature, pipette 3 mL sulphuric/acetic acid mixture into the sample, shake to ensure thorough mixing, and leave standing for 20 min. Now add 20 mL distilled water to the sample, and wash it into a separating funnel with a further 80 mL distilled water.
- Now add the extraction solution to the sample in the separating funnel, the first addition being 10 mL and subsequent additions being of 6 mL each at intervals of 1 min, shaking in between additions. All the extract so obtained is then filtered into the colorimetric cell.
- Carry out the measurement with the 662 Photometer at 555 nm.
- A blank control sample and 2 – 3 standard comparison samples (e.g. samples of known boron content) should be measured concurrently with each determination; the result is obtained by comparison with the blank control.

Calculation

The result is obtained by plotting a calibration curve.

Limits of determination

0.02 mg/L B

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Remarks

- This method has been developed primarily for the analysis of mineral waters. A calibration curve was plotted for 0.02 ... 0.4 mg/L B in the presence of 1000 mg Na^+, 1000 mg Ca^{2+} and 2 mg Fe^{2+}/L. The extinction values obtained are somewhat lower than would be the case with a pure boric acid solution.
- Samples containing more than 0.05 mg/L B must be diluted before analysis. The Lambert/Beer's law is valid over the range 0.02 ... 0.05 mg/L B.
- Fluorides vitiate the determination and must be removed by the Gaestel/Huré method.
- Oxidising agents also interfere with the determination, and therefore have to be reduced. Hayes and Metcalfe, and also Elwell and Wood, have described other sources of error (see literature references below).

Literature

- Thierig/Umland, Fresenius, Z. Anal. Chem, 211, 161 (1965)
- Hayes/Metcalfe, Analyst, 87, 956 (1962) and 88, 471 (1963)
- Elwell/Wood, Analyst, 88, 475 (1963)
- Gaestel/Huré, Bull. Soc. Chim. France, 16, 830 (1949)