

OIV-MA-AS323-03 Boron (Rapid Colorimetric Method)

Type IV method

1. Principle

The alcohol content of the wine is removed by reducing the volume by half by rotary evaporation. The wine is then passed through a column of polyvinylpyrrolidone, which retains the coloring agents. The eluate is collected quantitatively and the boron concentration determined by complexation with azomethine H at pH 5.2 followed by spectroscopic analysis at 420 nm.

2. Apparatus

2.1. Rotary evaporator

2.2. Spectrophotometer capable of measuring absorbance wavelengths between 300 and 700 nm

2.3. Cells of 1 cm optical path

Glass column of 1 cm internal diameter and 15 cm in length containing an 8 cm layer of polyvinylpyrrolidone.

3. Reagents

3.1. Azomethin H (4-hydroxy-5-(2-hydroxybenzylideneamino)-2,7-naphthalenedisulfonic acid)

3.2. Azomethin H solution

Place 1 g of azomethin H and 2 g of ascorbic acid in a 100 mL volumetric flask and add 50 mL double distilled water. Warm slightly to dissolve and make up to the mark with double distilled water. The reagent is stable for 2 days if kept cold.

3.3. Buffer solution pH 5.2

Dissolve 3g of EDTA (disodium salt of ethylenediaminetetraacetic acid) in 150 mL of double distilled water. Add 125 mL acetic acid ($\rho_{20} = 1.05 \text{ g/mL}$) and 250 g of ammonium acetate, $\text{NH}_4\text{CH}_3\text{COO}$, and dissolve. Check the pH with a pH meter and adjust if necessary to pH 5.2.

3.4. Boron stock standard solution, 100 mg/L

Use of a commercial standard solution is preferable. Alternatively this solution can be

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prepared by dissolving 0.571 g of boric acid, H_3BO_3 , dried beforehand at 50 °C until constant weight, in 500 mL double distilled water and made up to 1 liter.

3.5. Boron standard solution, 1 mg/L

Dilute the stock solution, 100 mg/L (3.4) 1/100 with double distilled water.

Polyvinylpolypyrrolidone or PVPP (see International Enological Codex)

4. Procedure

Eliminate alcohol from 50 mL of wine by concentration to half the original volume in a rotary evaporator at 40°C and make up to 50 mL with double distilled water.

Take 5 mL of this solution and pass it through the PVPP column (2.4). The coloring agents are completely retained. Collect the eluate and the rinsing waters from the column and place in a 50 mL volumetric flask and make up to the mark with water.

The colorimetric determination is performed in a volume of 5 mL of eluent placed in a 25 mL volumetric flask; dilute to approximately 15 mL with double distilled water and add the following (stirring after each addition):

- 5 mL of azomethin H solution (3.2)
- 4 mL of pH 5.2 buffer solution (3.3)

Make up to 25 mL with double distilled water.

Wait 30 min and determine the absorbance A_s , at 420 nm. The zero of the absorbance scale is set using distilled water.

Use a blank consisting of 5 mL of azomethin H solution and 4 mL of pH 5.2 buffer solution in 25 mL of double distilled water. Wait 30 min and read the absorbance A_b under the same conditions. The absorbance must be between 0.20 and 0.24; a higher absorbance demonstrates boron contamination in the water or the reagents.

Preparation of the calibration curve

In 25 mL volumetric flasks, place 1 to 10 g of boron, corresponding to 1 to 10 mL of boron standard solution 1 mg/L (3.5) and continue as indicated in 4.0. The calibration graph representing the net absorbance ($A_s - A_b$) in relation to the concentration is a straight line passing through the origin.

Where:

- A_s = absorbance of sample

A_b = absorbance of blank

5. Calculations

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The μg of boron contained in 5 mL of eluate, (corresponding to 0.5 mL of wine) obtained from interpolating the net absorbance values of $(A_s - A_b)$ on the calibration graph is E. The content, B, in milligrams of boron per liter is given by:

$$B \text{ mg/L} = \frac{E}{0.5}$$

Bibliography

- WOLF B., *Soil Science and Plant Analysis*, 1971, 2(5), 363-374 et 1974, 5(1), 39-44.
- CHARLOT C. and BRUN S., *F.V., O.I.V.*, 1983, no771.