

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 polyvinylpolypyrrolidone, 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 polyvinylpolypyrrolidone.

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.

Polyvinylpyrrolidone 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 azomethine 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 azomethine 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.