OIV-MA-AS315-01 Acetaldehyde

Type IV method

1. Principle

Acetaldehyde (ethanal) in carbon decolorized wine, reacts with sodium nitroferricyanide and piperidine and causes a green to violet color change whose intensity is measured at 570 nm.

2. Apparatus

Spectrophotometer permitting measurement of absorbance at a wavelength of 570 nm with a 1 cm optical cell path.

3. Reagents

3.1. Piperidine solution, ($C_5H_{11}N$) 10% (*v/v*).

Prepare just before use by mixing 2 mL of piperidine with 18 mL of distilled water.

3.2. Sodium nitroferricyanide solution, 0.4% (*m/v*).

In a 250 mL glass volumetric flask, dissolve 1 g of pulverized sodium nitroferricyanide, $Na_2[Fe(CN)_5\ NO].2H_2O$ in distilled water and make up to volume.

3.3. Activated carbon

3.4. Dilute hydrochloric acid, 25% (*v/v*)

3.5. Alkaline solution

Dissolve 8.75 g of boric acid in 400 mL sodium hydroxide solution, 1 M. Make up to 1 L with distilled water.

4. Procedure

1. Sample

Place approx. 25 mL of wine in a 100 mL Erlenmeyer flask, add 2 g of activated charcoal. Shake vigorously for a few seconds, allow to stand for 2 minutes and filter through a fluted slow filter to obtain a clear filtrate.

Place 2 mL of the clear filtrate into a 100 mL Erlenmeyer flask, add, while shaking, 5 mL of the sodium nitroferricyanide solution (3.2) and 5 mL of the piperidine solution (3.1). Mix and place the mixture immediately into a 1 cm optical cell. The coloration produced, which varies from green to violet, is measured with reference to air at a wavelength of 570 nm. This color change increases then decreases rapidly; measure

immediately and record the maximum value of the absorbance that is obtained after about 50 seconds. The concentration of acetaldehyde in the liquid analyzed is obtained using a calibration curve.

Note: If the liquid analyzed contains excess free acetaldehyde, it will be necessary, before beginning the total acetaldehyde determination, to first combine it with sulfur dioxide. To achieve this, add a small amount of excess free SO_2 to a portion of the liquid to be analyzed and wait for an hour before proceeding.

4.2. Preparation of the calibration curve

4.2.1. Solution of acetaldehyde combined with sulfur dioxide

Prepare a solution of between 5 to 6% (*m/v*) sulfur dioxide and determine the exact strength by titrating with 0.05 M iodine solution.

In a 1 L glass volumetric flask, add a volume of this solution which corresponds to 1500 mg of sulfur dioxide. Introduce into the flask, using a funnel, about 1 mL of acetaldehyde distillate recently distilled and collected in a cooling mixture. Make up to 1 liter with distilled water. Mix and allow to stand overnight.

The exact concentration of this solution is determined as follows:

Place in a 500 mL Erlenmeyer flask, 50 mL of the solution; add 20 mL of dilute hydrochloric acid (3.4) and 100 mL water. Titrate the free sulfur dioxide using a solution of 0.05 M iodine with starch as indicator, stopping at a faint blue end point. Add 100 mL of the alkaline solution, and the blue coloration will disappear. Titrate the combined sulfur dioxide and acetaldehyde with 0.05 M iodine until a faint blue end point is reached: let *n* be the volume used.

The acetaldehyde solution combined with SO_2 contains 44.05 *n* mg of acetaldehyde per liter.

4.2.2. Preparation of the calibration standards

In five 100 mL glass volumetric flasks, place respectively 5, 10, 15, 20 and 25 mL of the stock solution. Make up to volume with distilled water. These solutions correspond to acetaldehyde concentrations of 40, 60, 120, 160 and 200 mg/L. The exact concentration of the dilutions must be calculated from the acetaldehyde concentration of the stock solution (4.2.1) previously determined.

Proceed with the determination of acetaldehyde on 2 mL of each of these dilutions as indicated in 4.1. The graph of the absorbance of these solutions as a function of acetaldehyde content is a straight line that does not pass through the origin.

Bibliography

- REBELEIN H., *Dtsch. Lebensmit. Rdsch.,* 1970, 66, 5-6.