OIV-MA-AS322-04 Calcium

Type II method

1. Principle

Calcium is determined directly on diluted wine by atomic absorption spectrophotometry after the addition of an ionization suppression agent.

2. Apparatus

2.1. Atomic absorption spectrophotometer fitted with an air□acetylene burner.

2.2. Calcium hollow cathode lamp.

3. Reagents

1. Calcium standard solution 1 g/L. Use of a standard commercial calcium solution, 1 g/L, is preferred.

Alternatively this solution may be prepared by dissolving 2.5 g of calcium carbonate, $CaCO_3$, in sufficient hydrochloric acid (concentrated hydrochloric acid diluted 1:10) to dissolve it completely and making up to one liter with distilled water.

3.2. Dilute calcium standard solution, 50 mg/L

Note : Store the calcium solutions in polyethylene containers.

3.3. Dilute lanthanum standard solution, 50 g/L

Dissolve 13.369 g of lanthanum chloride, $LaCl_3.7H_2O$ in distilled water; add 1 mL, of dilute hydrochloric acid (concentrated hydrochloric acid diluted 1/10) and make up to 100 mL with distilled water.

4. Procedure

1. Preparation of sample

Place 1 mL of wine and 2 mL of the lanthanum chloride solution (3.3) in a 20 mL volumetric flask and make up to the mark with distilled water. The diluted wine contains 5 g lanthanum per liter.

Note: For sweet wines, 5 g lanthanum per liter is sufficient provided that the dilution reduces the sugar content to less than 2.5 g/L. For wines with higher concentrations of sugar, the lanthanum concentration should be increased to 10 g/L.

4.2. Calibration

Place 0, 5, 10, 15 and 20 mL, of dilute standard calcium solution (3.2) respectively into each of five 100 mL volumetric flasks, followed by 10 mL of the lanthanum chloride

solution (3.3) and make up to 100 mL with distilled water. The solutions prepared in this way contain 0, 2.5, 5.0, 7.5 and 10 mg of calcium per liter respectively, and each contains 5 g of lanthanum per liter. These solutions should be stored in polyethylene bottles.

4.3. Determination

Set the absorbance wavelength to 422.7 nm. Zero the absorbance scale using the zero standard (4.2). Aspirate the diluted wine directly into the spectrophotometer, followed in succession by the five standard solutions (4.2) and record the absorbance. Repeat each measurement.

5. Expression of results

1. Method of calculation

Plot a graph showing the variation in absorbance as a function of the calcium concentration in the standard solutions.

Record the mean value of the absorbance obtained with the sample of diluted wine on this graph and read its calcium concentration C. The calcium concentration in milligrams per liter of the wine to the nearest whole number is given by: 20 x C.

5.2. *Repeatability* (r)

- Concentration<60mg/L: r=2.7mg/L.
- Concentration > 60 mg/L: r = 4 mg/L.

3. *Reproducibility* (R)

- R mg/L = 0.114 x_i - 0.5.

where x_i = concentration in the sample in mg/L.