Method OIV-MA-BS-29 : R2009

Type IV method

Calcium- Determination by atomic absorption in spirit drinks of viti-vinicultural origin

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1. Principle

Calcium is determined by atomic absorption spectrophotometry with a reductive air acetylene flame using a calcium hollow-cathode lamp, wavelength of 422.7 nm, on the dealcoholised alcoholic beverage, concentrated 2 times. The measurement is performed in the presence of lanthanum chloride referred to as the "matrix modifier".

2. Appartus

2.1. Glassware

2.1.1. 25, 50, 100, 1000 ml volumetric flasks (class A).

2.1.2. 1, 2, 3, 4, 10, 50 ml volumetric pipettes (class A)

2.1.3. 100 ml test tube

2.1.4. 250 ml beaker (class A).

2.1.5. 20 ml tablet bottle

2.2. Spectrophotometer (sample setting for Varian 575 model)

2.2.1. Reducing air-acetylene flame, flow rates:

- air: : 7.5 Vmin
- C2 H2: 4.0 Vmin

2.2.2. Calcium hollow-cathode lamp with calcium; Wavelength: 422.7 nm, slit (slit): 0.2 nm, lamp intensity: 5 mA.

3. Reagents

3.1. Ultrapure demineralised water resistivity 18.2 M4.

3.2. Stock solution 2.3 to 1 g/l of Calcium: (e.g. Titrisol Merck).

3.3. Solution of 3.3 to 100 mg/1 of calcium

Place 10 ml of stock solution (3.2) in a 100 ml flask (2.1.1), fill to volume with demineralised water (3.1).

3.4. Hydrochloric acid d = 1.18 (35% minimum)

3.5. Lanthanum Chloride Solution, 25 g/1

Weigh 63.6 g of lanthanum chloride ($LaC1_3.6H_20$) in a 1000 ml flask

(2.1.1), add approximately 500 ml of demineralised water (3.1) then to the test tube (2.1.3) 50 ml of hydrochloric acid (3.4). After solubilisation, allow to cool and fill to volume with demineralised water (3.1).

3.6. Calibration range: 2, 4, 6, 8 mg/l of calcium.

Place successively 1.0, 2.0, 3.0, 4.0 ml of the solution at 100 mg/1Calcium (3.3) in four 50 ml vials (2.1.1), add 10 ml of the solution of lanthanum chloride (3.5), and fill to volume with demineralised water (3.1). Perform a blank test without calcium in the same conditions.

4. Sample preparation

The calcium content in alcoholic beverages is often very low, it is therefore necessary to concentrate the sample by evaporating the alcohol.

Pipette (2.1.2) 50 ml of the alcoholic beverage into a 250 ml beaker (2.1.4). Evaporate the alcohol in a water bath to about one volume of 10 ml. Leave to cool, then pour the concentrate into a vial of 25 ml (2.1.1), rinse the beaker and fill to volume with demineralised water (3.1).

Place 4 ml of this solution to be determined prepared in a clean, dry tablet bottle (2.1.5) with 1 ml of lanthanum chloride solution (3.5); cork, stir.

5. Determinations

Successively present the calibration solutions, the blank solution (3.6), and the samples (4.); ; note the corresponding absorbances.

Establish the calibration curve absorbance = f (concentration in mg/1 calcium) by the least squares method.

Deduce the concentration of calcium in mg/l taking into account the concentration factor.

6. Bibliography

1. Compendium of International methods of wine and must analysis, 1990, O.I.V. ed.