Method OIV-MA-BS-31 : R2009

Type IV method

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

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1. Principle

Iron is determined by atomic absorption spectrophotometry by means of an oxidising air-acetylene flame, using an iron hollow-cathode lamp, at a wavelength of 248.3 nm on the alcoholised alcoholic beverage.

2. Apparatus

2.1. Glassware.

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

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

2.1.3. 250 ml beaker (class A).

2.2. Spectrophotometer (sample setting for Varian 575 model)

2.2.1. Oxidising air-acetylene flame

- Flow rates: air:: 7.5 1/min
- C2 H2: 3.5 1/min

2.2.2. Iron hollow-cathode lamp; Wavelength: 248.3 nm, slit: 0.5 nm, lamp intensity: 5 mA.

3. Reagents

3.1. Ultrapure demineralised water resistivity 18.2 M□.m (e.g. Milli-Q).

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

3.3. Stock solution to 1 g/l of iron.

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. Calibration range: 2, 4, 6, 8 mg/1 of iron.

Place successively 1.0, 2.0, 3.0, 4.0 ml of the solution at 100 mg/1 of iron (3.30) in four 50 ml vials (2.1.1), fill to volume with demineralised water (3.1).

4. Sample preparation

4.1. Evaporation of alcohol.

Pipette (2.1.2) 50 ml of the alcoholic beverage in a 250 ml beaker (2.1.3). Evaporate the alcohol in a water bath to about one volume of 10 ml. Leave to cool, then pour the concentrate into a 50 ml vial (2.1.1), rinse the beaker and fill to volume with demineralised water (3.1).

Dilution in demineralised water (3.1) is only required if the concentration of iron is greater than 8 mg/l.

5. Determinations

Successively present the calibration solutions (3.4), and samples (4.1.);
note the corresponding absorbances.
Establish the calibration curve absorbance = f (concentration in mg/1 calcium) by the least squares method.
Deduce the concentration of iron (mg/1) taking into account any dilution.

6. Bibliography

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