COEI-2-CALCIU Determination of calcium by atomic absorption spectrometry

1. Principle

The calcium is directly determined in the liquid oenological product (or in the mineralisation solution) suitably diluted by atomic absorption spectrometry by air-acetylene flame after the addition of spectral buffer.

2. Apparatus

Instrumental parameters (given as an example)

- Atomic absorption spectrophotometer
- Reducing air-acetylene flame
- Hollow-cathode lamp (calcium)
- wave length: 422.7 nm
- width of slit: 0.2 nm
- intensity of the lamp: 5 mA
- No correction of non specific absorption.

3. Reagents

3.1. demineralised water

3.2. calcium reference solution at 1 g/l, commercial or prepared as follows: dissolve 5.8919 g of $Ca(NO_3)_2.4H_2O$ in a solution of HNO_3 0.5 M, adjust at 1 l with HNO_3 0.5 M.

3.3. calcium solution at 100 mg/l:

- place 10 ml of the reference solution in a 100 ml graduated flask and 1 ml of pure nitric acid.
- complete to volume with demineralised water

4. concentrated hydrochloric acid (R): 35% minimum

5. lanthanum solution at 25 g/l:

- weigh 65.9 g lanthanum chloride ($LaCl_3.6H_2O$) in a 250 ml cylindrical vase, transfer to a 1000 ml graduated flask with demineralised water; add to the test tube 50 ml of concentrated hydrochloric acid (R); after solubilisation, allow to cool, complete to volume with demineralised water.

6. set of calibration solutions: 0, 2, 4, 6, 8 mg/l of calcium

- place successively 0, 1,0, 2,0, 3,0 and 4.0 ml of the solution at 100 mg/l of calcium in 5, 50 ml graduated flasks, add 10 ml of lanthanum solution at 25 g/l, complete to volume with demineralised water.

4. Preparation of samples

4.1. Case of liquid or solution oenological products

In a 50 ml graduated flask place 10 ml of the lanthanum solution and a volume of sample as after having being completed to volume with demineralised water; the concentration is below 8 mg/l.

4.2. Case of solid oenological products

Proceed with mineralisation by dry process;

Put in each solution of the set the same quantity of acid used for putting cinders in solution or mineralisation (see chapter “Mineralisation”).

Take up cinders and 2 ml of concentrated hydrochloric acid (35% minimum) in a 100 ml flask; add 20 ml of lanthanum solution at 25 g/l and complete to volume with demineralised water.

Perform a blank test in the same conditions.

5. Procedure

Pass each solution of the set in ascending order of the concentration of calcium.

For each solution, perform 2 absorbance readings when they are perfectly stabilised (integration time of signal: 10 seconds).

Pass each sample twice and calculate the calcium content.