

## **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  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  in a solution of  $\text{HNO}_3$  0.5 M, adjust at 1 l with  $\text{HNO}_3$  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 ( $\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$ ) 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.