

after appropriate dilution, by the spectrometry of atomic absorption in the flame.

2- Equipment:

2-1- Glassware:

2-1-1- Volumetric flasks of class A of 25; 50; 100; 200; 250 and 500 ml.

2-1-2- Bulb pipettes of class A of 1; 2; 5 and 10 ml.

2-2- Spectrophotometry

2-2-1- Spectrophotometer of atomic absorption in the flame of Varian Spectra A 220

2-2-2- Instrumental parameters:

- Flame air -acétylène (Pair pressure 4 bar, C₂H₂ pressure: 1, 2 bar)

- Wavelength: 422.7 nm

- Slit width: 0.5 nm

- Lamp current: 10 mA

- With non-specific absorption correction.

3- REAGENTS

3.1) Distilled water (electric conductivity <3 μ S / cm)

3.2) Concentrated hydrochloric acid p.a d: 1.19

3.3) super-pure nitric acid 65%

3.4) p.a Hydrogen peroxide (30% v / v)

3.5) lanthanum solution at 50 g / l from the lanthanum oxide or lanthanum chloride

3.6) calcium stock solution at 1 g / l (Titrisol Merck) or equivalent

- Calcium solution 100 mg / l

Place 5 ml of the solution - mother (3.6) in a 50 ml flask (2.1.1), make up the volume with distilled water (3.1). Calibration range 1; 2 and 4 mg / l of calcium: Place successively. 1; 2 and 4 ml of the solution containing 100 mg / l (3.6.1 in 100 ml volumetric flasks (2.1.1). Add 1 ml of nitric acid (3.3) and 2 ml of lanthanum solution (3.5) up to volume with distilled water (3.1). Perform a blank without calcium in the same conditions.

1- The preparation of the samples

4-1 Sample containing organic compounds:

According to the content of the element to be determined, and to avoid potential dilution, if not necessary, place a sample test, weighing 0.01 mg in a capsule porcelain or platinum, dry in an oven at 105 ° C or in a hot plate (mild drying), then place the dish in a muffle furnace. Set the oven to reach 550 ° C gradually and let incinerate for 12 hours (sometimes less, depending on the product). Cool and add 5 to 10 ml of concentrated hydrochloric acid (3.2) and 3-5 drops of oxygenated water (3.4), heated on a hotplate until the ashes apparently dissolve more. Filter and recover in a volumetric flask whose volume V depends on the content in the research :

- If the content is > 0.1%) V - 250 or 500 ml.
- If the content is < 0.1%) V - 25: 50 or 100ml.

Prepare a blank sample under the same conditions

4-2- Sample containing no organic compounds

2- Determination:

Successively present the white and the calibration solutions (3.6.2). white there is a 1% solution of nitric acid (3.3) and 4% (v / v) of the lanthanum solution (3.5). Then present the blank sample and samples added to the 4% lanthanum solution (v / v), perform dilutions if necessary (eg If the sample reading is outside the calibration range). The device software provides the calcium concentration of samples in mg / l Calculation: The% calcium content: Music player (Ech) - Music player (B) .V .F

10.000 P.E

Music player (Ech): Sample Playback mg / l

Music player (B): Reading of blank sample in mg / l

V: Volume recovery after incineration in ml

P.E: Sample Test portion in grams.