COEI-2-PLOMB Determination of lead by atomic absorption spectrometry

1. Principle

After mineralisation of the sample in an acid medium, the lead is determined by spectrometry without flame (electro-thermal atomisation).

2. Apparatus

2.1. Instrumental parameters: (given as an example)

Atomic absorption spectrophotometer equipped with an atomiser with a graphite tube

- wave length: 283.3 nm
- hollow-cathode lamp (lead)
- width of slit: 0.5 nm
- intensity of the lamp: 5 mA
- correction of continuum: by Zeeman effect
- introduction in hot conditions of the samples in the graphite oven by an automatic distributor (rinsing water contains 2 drops of Triton per litre)
- measurement of signal: peak height
- time of measurement: 1 second
- number of measurements per sample: 2
- pyrolytic graphite tube
- pyrolytic graphite oven containing a platform of L'Vov
- tantalised (tantalisation of a platform: see above).

Parameters for oven

temperature (°C)	time (s)	gas flow rate (l / min)	type of gas	Reading of signal
150	20.0	3.0	argon	no
150	35.0	3.0	argon	no

800	15.0	3.0	argon	no
800	30.0	3.0	argon	no
800	2.0	0.0	argon	no
2250	0.8	0.0	argon	yes
2250	1.0	0.0	argon	yes
2500	1.0	1.5	argon	no
1200	9.0	3.0	argon	no
75	10.0	3.0	argon	no

2.2. Adjustments of the automatic sampler
(given as an example)

	volumes injected in µl		
	lead solution at 50 µg/l	blank	matrix modifier
blank	0	10	2
calibration N° 1	1	9	2
calibration N° 2	2	8	2
calibration N° 3	3	7	2
calibration N° 4	4	6	2
calibration N° 5	6	4	2
Sample to be measured	10	0	2

3. Reagents

3.1. Pure demineralised water for analysis

3.2. Pure nitric acid for analysis at 65%

3.3. Ammonium dihydrogenophosphate

3.4. Matrix modify: ammonium dihydrogenophosphate at 6%.

Introduce 3 g of ammonium dihydrogenophosphate in a 50 ml graduated flask, dissolve and complete to volume with demineralised water.

Lead reference solution at 1 g/l commercial or prepared as follows: dissolve 1.5985 g of pure $Pb(NO_3)_2$ for analysis in a solution of HNO_3 0.5 M, adjust at 1 l avec HNO_3 0.5 M.

Lead solution at 10 mg / l: place 1 ml of the reference lead solution at 1 g/l in a 100 ml graduated flask; add 1 ml of nitric acid at 65% complete to volume with pure demineralised water for analysis.

Lead solution at 0.1 mg/l: place 1 ml of the lead solution at 10 mg/l in a 100 ml graduated flask, add 1 ml of nitric acid at 65%; complete to volume with pure demineralised water for analysis.

Set of calibration solutions: 0, 50, 100, 150, 200, 300 µg/l of lead.

The automatic distributor cycle allows to directly inject these quantities of lead on the platform from the lead solution at 0.050 mg/l.

4. Preparation of samples

- The liquid or solution samples must have concentrations between 0 and 300 µg/l of lead.
- The solid samples will be mineralised by wet process (attack by nitric acid).
- The blank is made up of pure water for analysis containing 1% of nitric acid at 65%.

5. Procedure

The calibration curve represents the variations of absorbencies depending on the concentrations enabling to calculate the lead content of the samples.