

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 |

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| | | | | |
|------|------|-----|-------|-----|
| 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 $\mu\text{g/l}$ | 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

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- 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 $\text{Pb}(\text{NO}_3)_2$ 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 $\mu\text{g/l}$ 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 $\mu\text{g/l}$ 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.