COEI-2-NICKEL Determination of nickel by atomic absorption spectrometry

1. Principle

The nickel is directly determined by atomic absorption 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: 232.0 nm
- hollow-cathode lamp (nickel)
- width of the slit: 0.2 nm
- intensity of the lamp: 4 mA
- correction of continuum by the Zeeman effect
- Introduction in hot conditions of the samples in the graphite oven with an automatic distributor
- rinsing water contains 2 drops of Triton per litre.
- measurement of signal: peak height.
- Time of measurement: 1 second.
- pyrolytic graphite tube:
- pyrolytic graphite oven containing a platform of L'Vov tantalised.
- tantalisation of a platform: see above.
- inert gases: argon and argon + hydrogen mixture (95%: 5%).

Parameters for oven:

Parameters for oven for determining nickel

step n°	temperature (°C)	time (s)	gas flow rate (l/min)	type of gas	reading of signal
1	85	5.0	3.0	argon	no
2	95	40.0	3.0	argon	no
3	120	10.0	3.0	argon	no
4	800	5.0	3.0	argon	no
5	800	1.0	3.0	argon	no
6	800	2.0	0	argon	no
7	2 400	1.1	0	argon + hydrogen	yes
8	2 400	2.0	0	argon + hydrogen	yes
9	2 400	2.0	3.0	argon	no
10	75	11.0	3.0	argon	no

2.2. Adjustment of automatic sampler (given as an example)

- *Parameters of automatic sampler*

	volume injected in µl		
	solution of Ni at 50 µg/l	blank	matrix modifier
blank		17	3
calibration 1	5	12	3

calibration 2	10	7	3
calibration 3	15	2	3
sample	5	12	3

3. Reagents

3.1. Pure demineralised water for analysis
3.2. Pure nitric acid for analysis at 65%
3.3. Anhydrous palladium chloride (59% in Pd)
3.4. Pure hexahydrated magnesium nitrate for analysis
3.5. Ammonium dihydrogenophosphate
3.6. Matrix modifier: mixture of palladium chloride and magnesium nitrate (dissolve 0.25 g of $PdCl_2$ and 0.1 g of $Mg(NO_3)_2.6H_2O$ (3.4) in 50 ml of demineralised water) ammonium dihydrogenophosphate at 6% (dissolve 3 g de $NH_4H_2PO_4$ in 50 ml of demineralised water), (3.1).
3.7. L-ascorbic acid
3.8. Analytical blank solution: L-ascorbic acid solution at 1% (m/v).
3.9. Nickel reference solution at 1 g/l (1000 µg/ml) off the shelf or prepared as follows: dissolve 4.9533 of $Ni(NO_3)_2.6H_2O$ in a solution of HNO_3 0.5 M, adjust at 1 l with HNO_3 0.5 M.

4. Procedure

Nickel solution at 10 mg/l: place 1 ml of the reference solution (3.8) in a 100 ml graduated flask, add 5 ml of nitric acid (3.2); complete to volume with demineralised water.

Nickel solution at 50 µg/l: place 1 ml of the nickel solution at 10 mg/l in a 200 ml graduated flask, 10 ml of nitric acid (3.2) and complete with demineralised water.

Set of calibration solution: 0, 50, 100 and 150 µg/l of nickel.

The automatic distributor cycle enables to perform this calibration on the platform from a nickel solution at 50 µg/l.

5. Preparation of samples

5.1. Case of liquid or solution samples

No preparation or sample dilution is necessary; the samples are placed directly in the cups of the automatic injector.

5.2. Case of solid samples

The solid samples are mineralised by dry process.

6. Determinations

The calibration graph (absorbance depending on the concentration of nickel) gives the concentration of nickel in the samples.