

## **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*

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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 $\mu\text{l}$		
	solution of Ni at 50 $\mu\text{g/l}$	blank	matrix modifier
blank		17	3
calibration 1	5	12	3

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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<sub>2</sub> and 0.1 g of Mg(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (3.4) in 50 ml of demineralised water) ammonium dihydrogenophosphate at 6% (dissolve 3 g de NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> 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<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O in a solution of HNO<sub>3</sub> 0.5 M, adjust at 1 l with HNO<sub>3</sub> 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

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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.