COEI-2-SELENI Determination of selenium by atomic absorption spectrometry

1. **Principle**

After mineralisation of the sample by wet process, the selenium is determined by atomic absorption spectrometry without flame (electro-thermal atomisation in the graphite oven).

2. **Apparatus**

2.1. Glassware

- Graduated flasks 50, 100 ml (class A)
- Graduated pipettes 1, 5 and 10 ml (class A)
- Polypropylene tubes 50 ml with screw top.

2. Instrumental parameters**:** (given as an example)

- Atomic absorption spectrophotometer equipped with an atomiser
- with a graphite tube.
- wave length: 196.0 nm
- hollow-cathode lamp (selenium)
- width of slit: 1.0 nm.
- intensity of the lamp: 10 mA
- correction of continuum by the Zeeman effect
- introduction in hot conditions of the samples in the graphite oven with anautomatic distributor (rinsing water contains 2 drops of Tritonper 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 given procedure beforehand.
inert gas: argon.
parameters for oven: table I

Table I - Parameters for oven for determining selenium

step	temperature (°C)	time (s)	gas flow rate (l/min)	type of gas	reading of signal
1	85	5	3.0	argon	no
2	95	40	3.0	argon	no
3	120	10	3.0	argon	no
4	1 000	5	3.0	argon	no
5	1 000	1	3.0	argon	no
6	1 000	2	0	argon	no
7	2 600	0.8	0	argon	yes
8	2 600	2	0	argon	yes
9	2 600	2	3.0	argon	no

2.3. Automatic sampler parameters (table II)
(given as an example)

Table II - Parameters de automatic sampler.

	volumes injected in µl		
	solution	blank	matrix modifier
blank		17	3
calibration n°1 50 µg/l	5	12	3

calibration n°2 100 µg/l	10	7	3
calibration n°3 150 µg/l	15	2	3
sample	15	2	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 $Mg(NO_3)_2.6H_2O$ in 50 ml of demineralised water) ammonium dihydrogenophosphate at 6% (dissolve 3 g de $NH_4H_2PO_4$ in 50 ml of demineralised water).

3.7. Selenium reference solution at 1 g/l, off the shelf or prepared as follows: dissolve 1.4052 g SeO_2 in a solution of HNO_3 0.5 M, adjust at 1 l avec HNO_3 0.5 M.

3.8. Selenium solution at 10 mg/l: place 1 ml of the reference solution at 1 g/l in a 100 ml graduated flask; add 5 ml of nitric acid at 65%; complete to volume with pure demineralised water for analysis

3.9. Selenium solution at 50 µg/l: place 0.5 ml of the selenium solution at 10 mg/l, 5 ml of nitric acid at 65% in a 100 ml graduated flask; complete to volume with pure demineralised water for analysis.

3.10. Set of calibration solutions: 0, 50, 100 and 150 µg/l of selenium.

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

4. Preparation of samples

Weigh with precision a test sample of 1 to 3 g in the graduated tube; add 5 ml of nitric acid at 65%; close with the screw cap; leave 12 hours at room temperature;

place the tube in a water bath at 90°C for 3 hours (the caps are unscrewed during the heating); allow to cool; adjust the volume to 20 ml with pure demineralised water for analysis.

5. Determinations

Set up the calibration graph (absorbance depending on the concentration in µg/l of selenium); determine the concentration of selenium in the samples.

Calculate the concentration of selenium in the mineralisate, then in the samplein µg/kg.