COEI-2-CADMIU Determination of cadmium by atomic absorption spectrometry

1. Principle

The cadmium is determined in solid oenological products after mineralisation by wet process or directly for liquid oenological products or put in a solution.

The determinations are performed by atomic absorption without a flame (electro-thermal atomisation in a graphite oven).

2. Apparatus

2.1. Instrumental parameters (given as an example)

Spectrophotometer equipped with an atomiser with a graphite tube.

- wave length: 228.8 nm
- hollow-cathode lamp (cadmium)
- width of slit: 1 nm
- intensity of the lamp: 3 mA
- correction of continuum by the Zeeman effect
- graphite oven with a tantalised platform
- (tantalisation procedure of the platform described above)
- adjusting the oven for an analysis:

step	temperature (°C)	time (s)	gas flow rate (/ mn	type of gas	reading of signal
1	100	35	3.0	argon	no
2	500	10	3.0	argon	no
3	500	45	1.5	argon	no
4	500	1	0.0	argon	no
5	2250	1	0.0	argon	yes

6	2250	1	0.0	argon	yes
7	2500	2	1.5	argon	no
8	1250	10	3.0	argon	no
9	75	10	3.0	argon	no

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

	volumes injected in µl		
	solution of Cd at 8 µg/l	blank	matrix modifier
blank	0	10	2
calibration N° 1 at 8 µg / l	1	9	2
calibration N° 2 at 16 µg / l	2	8	2
calibration N° 3 at 24 µg / l	3	7	2
calibration N° 4 at 32 µg / l	4	6	2
Sample to be dosed	5	5	2

3. Reagents

- Demineralised water
- Pure nitric acid for analysis at 65%
- Anhydrous palladous chloride (59% in Pd)
- Magnesium nitrate with 6 water molecules (ultra pure)
- Ammonium dihydrogenophosphate

Matrix modifier: palladous chloride and magnesium nitrate mixture (dissolve 0.25 g of $PdCl_2$ and 0.1 g of $Mg(NO_3)_2.6H_2O$ in 50 ml of demineralised water) or ammonium dihydrogenophosphate at 6% (dissolve 3 g of $NH_4H_2PO_4$ in 50 ml of demineralised

water).

Cadmium reference solution at 1 g/l, commercial or prepared as follows: dissolve 2.7444 g $Cd(NO_3)_2.4H_2O$ in a solution of HNO_3 0.5 M, adjust to 1 l with HNO_3 0.5 M.

Cadmium solution at 10 mg/l: place 1 ml of the reference solution in a 100 ml graduated flask, add 5 ml of pure nitric acid and complete to volume with demineralised water.

Cadmium solution at 0.8 g/l: place 4 ml of the diluted solution in a 50 ml graduated flask, add 2.5 ml of pure nitric acid and complete to volume with demineralised water.

Calibration range at 0, 8, 16, 24 and 32 µg/l of cadmium.

4. Preparation of samples

No preparation is necessary for liquid oenological products or in solution form; solid products are mineralised by wet process.

The blank solution is made up of a pure nitric acid solution for analysis at 1%.

5. Procedure

Each calibration solution is passed right after the blank solution. Perform 2 successive absorbance readings and establish the calibration curve.

Calculate the cadmium content of the samples while taking into account the test sample of different dilutions.