

## 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

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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 $\mu\text{l}$		
	solution of Cd at $8 \mu\text{g/l}$	blank	matrix modifier
blank	0	10	2
calibration N° 1 at $8 \mu\text{g/l}$	1	9	2
calibration N° 2 at $16 \mu\text{g/l}$	2	8	2
calibration N° 3 at $24 \mu\text{g/l}$	3	7	2
calibration N° 4 at $32 \mu\text{g/l}$	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  $\text{PdCl}_2$  and 0.1 g of  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in 50 ml of demineralised water) or ammonium dihydrogenophosphate at 6% (dissolve 3 g of  $\text{NH}_4\text{H}_2\text{PO}_4$  in 50 ml of demineralised

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water).

Cadmium reference solution at 1 g/l, commercial or prepared as follows: dissolve 2.7444 g  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  in a solution of  $\text{HNO}_3$  0.5 M, adjust to 1 l with  $\text{HNO}_3$  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  $\mu\text{g}/\text{l}$  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.