

## 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

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dihydrogenophosphate at 6% (dissolve 3 g of  $\text{NH}_4\text{H}_2\text{PO}_4$  in 50 ml of demineralised 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  $\mu\text{g/l}$  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.