# 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.