COEI-2-CHROME Determination of chrome by atomic absorption spectrometry

1. Principle

The chrome is determined by atomic absorption spectrophotometer without flame.

2. Apparatus

2.1. Experimental parameters (given as an example)

- Atomic absorption spectrophotometer
- wave length: 357.9 nm
- hollow-cathode lamp (Chrome)
- width of slit: 0.2 nm
- intensity of the lamp: 7 mA
- correction of continuum by the Zeeman effect
- introduction in hot conditions of the samples in the graphite oven
- measurement of the signal: peak height
- time of measurement: 1 second
- number of measurements per sample: 2
- pyrolytic graphite tube:
- pyrolytic graphite oven containing a platform L'Vov tantalised
- tantalisation of platform (see above) inert gas: argon - hydrogen mixture (95%; 5%)
- parameters for oven:

step	temperature (°C)	time (s)	gas rate flow (l / mn)	type of gas	reading of signal
1	85	5	3.0	argon + hydrogen	no
2	95	40	3.0	argon + hydrogen	no

3	120	10	3.0	argon + hydrogen	no
4	1000	5	3.0	argon + hydrogen	no
5	1000	1	3.0	argon + hydrogen	no
6	1000	2	0.0	argon + hydrogen	no
7	2600	1.2	0.0	argon + hydrogen	yes
8	2600	2	0.0	argon + hydrogen	yes
9	2600	2	3.0	argon + hydrogen	no
10	75	11	3.0	argon + hydrogen	no

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

	volumes injected in µl		
	chrome solution at 50 µg/l	blank	matrix modifier
blank	0	17	3
calibration N° 1 at 50 µg/l	5	12	3
calibration N° 2 at 100 µg/l	10	7	3
calibration N° 3 at 150 µg/l	15	2	3
sample to be measured	5	12	3

3. Reagents

3.1. pure demineralised water for analysis

3.2. pure nitric acid for analysis at 65%

3.3. anhydrous palladous 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 of $NH_4H_2PO_4$ in 50 ml of demineralised water).

3.7. Reducing agent: L-ascorbic acid in solution at 1% m/v.

3.8. chrome reference solution at 1 g/l, commercial or prepared as follows: dissolve 7.6952 g of $Cr(NO_3)_{3.}9H_2O$ in a solution of HNO_3 0.5 M, adjust at 1 l with HNO_3 0.5 M

3.9. chrome solution at 10 mg/l: place 1 ml of the reference solution in a 100 ml graduated flask, add 5 ml of nitric acid at 65% and complete to volume with demineralised water.

3.10. set of calibration solutions: 0, 50, 100 and 150 µg/l of chrome (see table: adjustments of the automatic sampler).

4. Preparation of samples

4.1. Case of liquid or solution oenological products

The preparations are performed manually or automatically by the diluter by following the data from the table “adjustments of the automatic sampler”.

4.2. Case of solid oenological products

Proceed with mineralisation by wet process. Do a blank test.

5. Procedure

Pass each solution of the set in ascending order of the concentration of chrome;

Pass each sample twice and calculate the chrome content while taking into account the test sample.