

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

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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 $\mu\text{g/l}$	blank	matrix modifier
blank	0	17	3
calibration N° 1 at 50 $\mu\text{g/l}$	5	12	3
calibration N° 2 at 100 $\mu\text{g/l}$	10	7	3
calibration N° 3 at 150 $\mu\text{g/l}$	15	2	3
sample to be measured	5	12	3

3. Reagents

3.1. pure demineralised water for analysis

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- 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 $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 50 ml of demineralised water) ammonium dihydrogenophosphate at 6% (dissolve 3 g of $\text{NH}_4\text{H}_2\text{PO}_4$ 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 $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ 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 $\mu\text{g}/\text{l}$ 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.