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International Standard ISO 7627/6

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Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 6 : Determination of chromium in contents from 0,01 to 2 % (m/m)

Métaux-durs — Analyse chimique par spectrométrie d'absorption atomique dans la flamme — Partie 6 : Dosage du chrome à des teneurs comprises entre 0,01 *et* 2 % (m/m)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Hardmetals — Chemical analysis by flame atomic absorption spectrometry —
Part 6 : Determination of chromium in contents from 0,01 to 2 % (m/m)

1 Scope and field of application

This part of ISO 7627 specifies the method to be used for the determination of the chromium content of hardmetals within the range 0,01 to 2 % (m/m) by flame atomic absorption spectrometry.

General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in ISO 7627/1.

2 Reference

ISO 7627/1, *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 1 : General requirements.*

3 Reagents

3.1 Potassium pyrosulfate.

3.2 Perchloric acid, ϱ 1,54 or 1,67 g/ml.

3.3 Ammonium citrate, solution.

Dissolve 100 g of citric acid in 1 500 ml of water and add 400 ml of ammonia solution (ϱ 0,91 g/ml).

3.4 Hydrogen peroxide, 30 % (m/m).

3.5 High purity stock solution, for calibration purposes, containing 1,000 g of chromium per litre.

NOTE — This value is understood to establish a maximum limit of 1,000 5 g and a minimum limit of 0,999 5 g.

4 Procedure

4.1 Test portion

Weigh, to the nearest 0,001 g, the relevant amount of the test sample indicated in table 1. Transfer it to a 100 ml conical flask (preferably of quartz).

NOTE — In this special case, the sample shall pass a 0,18 mm sieve.

4.2 Dissolution of the test portion

Add 5 g of the potassium pyrosulphate (3.1) and a few drops of the perchloric acid (3.2) to the beaker containing the test portion (4.1) and heat gently until the test portion is completely dissolved. Add 40 ml of the ammonium citrate solution (3.3), and about 0,5 ml of the hydrogen peroxide (3.4). Then transfer the solution to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

Table 1 — Test portion, instrumental parameters and characteristics of calibration functions

Content	Test portion	Dilution volume (V) for the test portion[1]	Oxidant	Wavelength	Reciprocal sensitivity, for 1 % absorption[1]	Linear range[1]
%	g	ml		nm	µg/ml	%
0,01 to 0,1	0,500	100	N_2O	357,9	0,11	0 to 0,12
0,1 to 0,5	0,500	500	N_2O	357,9	0,11	0,1 to 0,5
0,1 to 0,5	0,100	100	N_2O	357,9	0,11	0,1 to 0,5
0,5 to 2	0,100	500	N_2O	357,9	0,11	0,4 to 2

1) Guidelines for information only.

4.3 Dilution volume

Prepare the relevant dilution volume for the analysis according to table 1 as follows :

4.3.1 Dilution volume 100 ml : use the solution in 4.2.

4.3.2 Dilution volume 500 ml : transfer 20 ml of the solution in 4.2 to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

4.4 Preparation of calibration and blank solutions

4.4.1 Prepare at least six solutions according to 4.2 with a matrix composition as similar as possible to the test portion to be analysed, but without making up to volume. Then add increasing volumes of the properly diluted stock solution of chromium (3.5) according to the concentration range to be covered. Make up to 100 ml and mix.

4.4.2 Also prepare calibration solutions with a diluted matrix in accordance with 4.3 for dilution volumes of 500 ml, if necessary.

4.4.3 Prepare a blank solution (see 4.4.1) without the addition of chromium.

4.5 Adjustment of the atomic absorption spectrometer

SAFETY PRECAUTION : Follow the manufacturer's recommendation on igniting and extinguishing the flame.

Optimize the response of the instrument at the wavelength given (see table 1).

Preheat the burner for about 5 min and then adjust the fuel and correct the burner to obtain maximum absorption while aspirating a calibration solution. Make sure that the absorbance reading is not drifting. Aspirate water and set the initial reading to zero absorbance.

4.6 Atomic absorbance measurements

4.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the readings. Aspirate water between each solution. Make at least two measurements for each solution. If a build-up of solids occurs on the burner slit, it will cause a decrease of sensitivity and must be removed.

4.6.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration, in milligrams per litre, of chromium.

4.6.3 Convert the absorbance values of the test solutions corrected for the blank to milligrams of chromium per litre by means of the calibration curve.

5 Expression of results

5.1 Calculation

The chromium content, expressed as a percentage by mass, is given by the formula

$$\frac{c \times V}{10^4 \times m}$$

where

c is the concentration, in milligrams per litre, of chromium in the test solution;

V is the dilution volume, in millilitres;

m is the mass, in grams, of the test portion.

5.2 Permissible tolerances

The deviations between three independent determinations shall not exceed the values shown in table 2.

Table 2 — Permissible tolerances

Content % (m/m)	Range % (m/m)
from 0,01 to 0,20	0,012
over 0,20 to 2	0,06 × (content)

5.3 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest value as shown in table 3.

Table 3 — Rounding of results

Content % (m/m)	Round to the nearest % (m/m)
from 0,01 to 0,20	0,01
over 0,20 to 2	0,05

If the chromium content is below 0,01 % (m/m), report it as less than 0,01 % (m/m).