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**Hårdmetall — Kemisk analys med flamatom-
absorptionsspektrometri — Del 6:
Bestämning av kromhalt mellan 0,01 och
2 % (m/m) (ISO 7627-6:1985)**

**Hardmetals — Chemical analysis by flame
atomic absorption spectrometry — Part 6:
Determination of chromium in contents
from 0,01 to 2 % (m/m) (ISO 7627-6:1985)**

Europastandarden EN 27 627-6:1993 gäller som svensk standard.

The European Standard EN 27 627-6:1993 has the status of a Swedish Standard.

Detta dokument innehåller den officiella engelska versionen av EN 27 627-6:1993.

This document contains the official English version of EN 27 627-6:1993.

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English version

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

(ISO 7627-6 : 1985)

Métaux-durs — Analyse chimique par spectrométrie d'absorption atomique dans la flamme — Partie 6: Dosage du chrom à des teneurs comprises entre 0,01 et 2 % (m/m) (ISO 7627-6 : 1985)

Hartmetalle — Chemische Analyse durch Flammenatomabsorptionsspektrometrie — Teil 6: Bestimmung des Chromgehaltes von 0,01 bis 2 % (m/m) (ISO 7627-6 : 1985)

This European Standard was approved by CEN on 1993-04-02. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

In 1992, ISO 7627-6 : 1985 *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 6: Determination of chromium in contents from 0,01 to 2 % (m/m)* was submitted to the CEN Primary Questionnaire procedure.

Following the positive result of the CEN/CS Proposal ISO 7627-6 : 1985 was submitted to the CEN Formal Vote. The result of the Formal Vote was positive.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 1993, and conflicting national standards shall be withdrawn at the latest by October 1993.

According to the Internal Regulations of CEN/CENELEC, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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 g	Dilution volume (V) for the test portion ¹⁾ ml	Oxidant	Wavelength nm	Reciprocal sensitivity, for 1 % absorption ¹⁾ µg/ml	Linear range ¹⁾ %
0,01 to 0,1	0,500	100	N ₂ O	357,9	0,11	0 to 0,12
0,1 to 0,5	0,500	500	N ₂ O	357,9	0,11	0,1 to 0,5
0,1 to 0,5	0,100	100	N ₂ O	357,9	0,11	0,1 to 0,5
0,5 to 2	0,100	500	N ₂ O	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).