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Handläggande organ

MATERIALNORMCENTRALEN

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**Hårdmetall — Kemisk analys med flamatom-
absorptionsspektrometri — Del 3: Bestäm-
ning av kobolt-, järn-, mangan- och nickel-
halt mellan 0,01 och 0,5 % (m/m)
(ISO 7627-3:1983)**

**Hardmetals — Chemical analysis by flame
atomic absorption spectrometry — Part 3:
Determination of cobalt, iron, manganese
and nickel in contents from 0,01 to 0,5 %
(m/m) (ISO 7627-3:1983)**

Europastandarden EN 27 627-3:1993 gäller som svensk standard.

The European Standard EN 27 627-3:1993 has the status of a Swedish Standard.

Detta dokument innehåller den officiella engelska versionen av EN 27 627-3:1993.

This document contains the official English version of EN 27 627-3:1993.

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Descriptors: Powder metallurgy, hard metals, chemical analysis, determination of content, transition metals, cobalt, iron, manganese, nickel, spectrometric analysis, atomis absorption spectrophotometry

English version

Hardmetals — Chemical analysis by flame atomic
absorption spectrometry — Part 3: Determination of
cobalt, iron, manganese and nickel in contents from 0,01
to 0,5 % (m/m)

(ISO 7627-3 : 1983)

Métaux-durs — Analyse chimique par
spectrométrie d'absorption atomique dans la
flamme — Partie 3: Dosage du cobalt, du fer,
du manganèse et du nickel à des teneurs
comprises entre 0,01 et 0,5 % (m/m)
(ISO 7627-3 : 1983)

Hartmetalle — Chemische Analyse durch
Flammenatomabsorptionsspektrometrie —
Teil 3: Bestimmung des Cobalt-, Eisen-,
Mangan- und Nickelgehaltes von 0,01 bis
0,5 % (m/m)
(ISO 7627-3 : 1983)

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.

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CEN

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Page 2
EN 27627-3 : 1993

Foreword

In 1992, ISO 7627-3 : 1983 *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 3 : Determination of cobalt, iron, manganese and nickel in contents from 0,01 to 0,5 % (m/m)* was submitted to the CEN Primary Questionnaire procedure.

Following the positive result of the CEN/CS Proposal ISO 7627-3 : 1983 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, United Kingdom.

NOTE. The European references to international publications are given in annex ZA (normative).

1 Scope and field of application

This part of ISO 7627 specifies the method to be used for the determination of cobalt, iron, manganese and nickel contents of hardmetals within the range 0,01 to 0,5 % (*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 Hydrofluoric acid, ρ 1,12 g/ml.

3.2 Nitric acid, ρ 1,42 g/ml.

3.3 Ammonium fluoride, 0,1 g/ml solution.

3.4 Caesium chloride, 0,01 g/ml solution.

3.5 High-purity stock solutions, for calibration purposes for each element to be determined, containing 1,000 g of the element 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, approximately 1 g of the test sample. Transfer it to a 100 ml polytetrafluorethylene beaker or a beaker of other suitable material. Cover the beaker.

4.2 Dissolution of the test portion

Add 10 ml of water, 5 ml of the hydrofluoric acid (3.1), and then 5 ml of the nitric acid (3.2), drop by drop, to the beaker containing the test portion (4.1) and heat gently until the test portion is completely dissolved. Add 10 ml of the caesium chloride solution (3.4) and 10 ml of the ammonium fluoride solution (3.3). Then transfer the solution totally to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

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 The concentration of the solution may be reduced by a factor of 10 for instruments of higher sensitivity by transferring 10 ml of the solution in 4.2 to a 100 ml polypropylene one-mark volumetric flask. Add 10 ml of the caesium chloride solution (3.4). Add 10 ml of the ammonium fluoride solution (3.3) 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 properly diluted stock solutions of the elements to be determined according to the concentration ranges 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 1 000 ml, if necessary.

Table 1 – Instrumental parameters and characteristics of calibration functions

| Element | Dilution volume (<i>V</i>) for 1 g test portion ¹⁾ ml | Oxidant | Wavelength nm | Reciprocal sensitivity, for 1 % absorption ¹⁾ µg/ml | Linear range ¹⁾ % | Notes |
|---------|--|------------------|------------------|--|---------------------------------|-------|
| Co | 100 | N ₂ O | 240,7 | 0,7 | 0,007 to 0,5 | 1 |
| Fe | 100 | N ₂ O | 248,3 | 0,3 | 0,003 to 0,05 | |
| | 1 000 | N ₂ O | 248,3 | 0,3 | 0,03 to 2,0 | |
| Mn | 100 | N ₂ O | 279,8 | 0,1 | 0,001 to 0,03 | 2 |
| | 1 000 | N ₂ O | 279,8 | 0,1 | 0,01 to 2,0 | 2 |
| Ni | 100 | N ₂ O | 232,0 | 0,3 | 0,003 to 0,05 | 3 |
| | 1 000 | N ₂ O | 232,0 | 0,3 | 0,05 to 5,0 | 3 |

1) Guidelines for information only.

NOTES

- When a broad spectral band pass is used, a single-element hollow cathode lamp is mandatory.
- Use of the triplet.
- Sensitivity is greatly dependent on matrix composition. Use of a narrow-spectral band pass and a single-element hollow-cathode lamp is mandatory.

4.4.3 Prepare one or more blank solutions (see 4.4.1) without the addition of the relevant element to be determined.

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 for the element being determined. 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. Solids which build up on the burner slit must be removed, otherwise they will cause a decrease of sensitivity.

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 the element.

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

5 Expression of results

5.1 Calculation

The element 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 the element 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 0,2 times the element content in per cent (absolute value).

5.3 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest value as shown in table 2.

Table 2 – Rounding of results

| Content % (<i>m/m</i>) | Round to the nearest % (<i>m/m</i>) |
|-----------------------------|--|
| from 0,01 to 0,1 | 0,01 |
| over 0,1 to 0,5 | 0,05 |

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