SLOVENSKI STANDARD

SIST EN 14941:2006

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Baker in bakrove zlitine ï Določ evanje kobalta ï Metoda FAAS

Copper and copper alloys - Determination of cobalt content - FAAS method

Kupfer und Kupferlegierungen - Bestimmung des Cobaltgehaltes - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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Copper and copper alloys - Determination of cobalt content - FAAS method

Cuivre et alliages de cuivre - Dosage du cobalt - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des Cobaltgehaltes - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

This European Standard was approved by CEN on 15 May 2006.

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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 14941:2006) has been prepared by Technical Committee CEN/TC 133 “Copper and copper alloys”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to prepare the following standard:

EN 14941, *Copper and copper alloys — Determination of cobalt content — FAAS method*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the cobalt content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having cobalt mass fractions between 0,010 % and 3,0 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1811-1, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products*

ISO 1811-2, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings*

NOTE Informative references to documents used in the preparation of this standard, and cited at the appropriate places in the text, are listed in the Bibliography.

3 Principle

Dissolution of a test portion in hydrochloric-nitric acid mixture followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 240,7 nm line emitted by a cobalt hollow-cathode lamp.

4 Reagents and Materials

4.1 General

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.2 Hydrochloric acid, HCl (ρ = 1,19 g/ml).

4.3 Nitric acid, HNO_3 (ρ = 1,40 g/ml).

4.4 Nitric acid solution, 1 + 1

Dilute 100 ml of nitric acid (4.3), in 100 ml of water.

4.5 Cobalt stock solution, 1,000 g/l Co

Weigh (1 ± 0,001) g of cobalt (Co ≥ 99,9 %) and transfer it into a 250 ml beaker. Add 20 ml of nitric acid solution (4.4), cover and heat gently until the cobalt is completely dissolved. Boil the solution for a few minutes to expel nitrous fumes. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of Co.

4.6 Cobalt standard solution, 0,100 g/l Co

Using a pipette, transfer 25,0 ml of cobalt stock solution (4.5) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare the solution immediately prior to use.

1 ml of this solution contains 0,100 mg of Co.

4.7 Lanthanum(III) chloride solution (100 g/l)

Dissolve 100 g of lanthanum(III) chloride ($LaCl_3 \cdot 7H_2O$) in a 600 ml beaker with water and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

4.8 Copper base solution, 20 g/l Cu

Weigh 10,0 g cobalt-free copper (Co ≤ 0,000 2 %) and transfer it into a 600 ml beaker. Add 200 ml of hydrochloric acid (4.2) and, cautiously, 200 ml of nitric acid solution (4.4). Heat until the copper is dissolved and then boil until nitrous fumes have been expelled. Cool to room temperature and transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Atomic absorption spectrometer, fitted with an air/acetylene burner.

5.3 Cobalt hollow-cathode lamp.

6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

Test samples shall be in the form of fine drillings, chips or millings with a maximum thickness of 0,5 mm.

7 Procedure

7.1 Preparation of the test portion solution

7.1.1 Test portion

Weigh (1 ± 0,001) g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker and cover with a watch glass. Add 20 ml of hydrochloric acid (4.2) and 20 ml of nitric acid solution (4.4). Heat gently until the test portion is completely dissolved, then continue heating to the boiling point until the nitrous fumes have been expelled. Wash the cover and the sides of the beaker with water and allow to cool. Transfer the solution to a 100 ml one-mark volumetric flask, add 10 ml of lanthanum(III) chloride solution (4.7), dilute to the mark with water and mix well.

Select an aliquot portion from the test portion solution according to the expected cobalt mass fraction and prepare a dilution in a volumetric flask as indicated in Table 1. Add lanthanum(III) chloride solution as shown in Table 1.

Table 1 — Dilution

Cobalt mass fractions %	Aliquot of the test portion solution (7.1.2) ml	Lanthanum(III)-chloride solution (4.7) ml	Final volume of diluted solution ml	Dilution factor D_r
0,01 to 0,2	100	0	100	1
0,1 to 1,0	20	8	100	5
0,5 to 3,0	20	48	500	5a × 5b[a]

[a] For explanation of dilution factors see the notes to Clause 8 Expression of results.

7.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents as used for the determination, but omitting the test portion.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of a standard material or a synthetic sample containing a known amount of cobalt and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

In all cases, copper, chloride and nitrate concentrations and acidity in the calibration solutions shall be similar to those of the test portion solutions.

The presence of copper in the standard calibration solutions compensates for chemical interaction effects of copper in the test portion solution. Normally no similar additions are required to compensate for the effect of alloying elements. If an alloying element is present in the material to be analysed in mass fraction > 10 %, an appropriate mass of this element shall be added to the calibration solutions.

The Co concentration of the calibration solutions shall be adjusted to suit the sensitivity of the apparatus used, so that the curve of absorbance as a function of the concentration is a straight line.

7.4.1.2 Calibration range for cobalt mass fractions between 0,01 % and 3,0 %

Into each of a series of eight 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.6) and of copper base solution (4.8) shown in Tables 2, 3 or 4, depending on the expected cobalt content. Introduce also, 10 ml of lanthanum(III) chloride solution (4.7). Dilute to the mark with water and mix well.

Table 2 — Calibration for cobalt mass fractions between 0,01 % and 0,20 %

Cobalt standard solution volume (4.6) ml	Corresponding cobalt mass mg	Corresponding cobalt concentration after final dilution mg/ml	Copper base solution volume (4.8) ml	Corresponding copper mass g	Corresponding cobalt mass fraction of sample %
0[a]	0	0	50	1,000	0
1	0,1	0,001	50	1,000	0,01
2	0,2	0,002	50	1,000	0,02
5	0,5	0,005	50	1,000	0,05
7	0,7	0,007	50	1,000	0,07
10	1,0	0,010	50	1,000	0,10
15	1,5	0,015	50	1,000	0,15
20	2,0	0,020	50	1,000	0,20

[a] Blank test on reagents for calibration curve.

Table 3 — Calibration for cobalt mass fractions between 0,05 % and 1,0 %

Cobalt standard solution volume (4.6) ml	Corresponding cobalt mass mg	Corresponding cobalt concentration after final dilution mg/ml	Copper base solution volume (4.8) ml	Corresponding copper mass g	Corresponding cobalt mass fraction of sample %
0[a]	0	0	10	0,200	0
1	0,1	0,001	10	0,200	0,05
2	0,2	0,002	10	0,200	0,10
5	0,5	0,005	10	0,200	0,25
7	0,7	0,007	10	0,200	0,35
10	1,0	0,010	10	0,200	0,50
15	1,5	0,015	10	0,200	0,75
20	2,0	0,020	10	0,200	1,00

[a] Blank test on reagents for calibration curve.