SLOVENSKI STANDARD

SIST EN 14939:2006

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Baker in bakrove zlitine ï Določ evanje berilija ï Metoda FAAS

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

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

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

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

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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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Foreword

This document (EN 14939:2006) 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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.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 14939, *Copper and copper alloys — Determination of beryllium 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 beryllium content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having beryllium mass fractions between 0,01 % 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 a nitrous oxide/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 234,8 nm line emitted by a beryllium hollow-cathode lamp.

4 Reagents and materials

WARNING — Beryllium is an extremely poisonous metal and the inhalation of dust can cause cancer.

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 Hydrochloric acid solution, 1 + 1

Dilute 1 000 ml of hydrochloric acid (4.2) in 1 000 ml of water.

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

4.5 Nitric acid solution, 1 + 1

Dilute 1 000 ml of nitric acid (4.4) in 1 000 ml of water.

4.6 Beryllium stock solution, 1,000 g/l Be

Weigh (1 ± 0,001) g of beryllium (Be ≥ 99,9 %) and transfer it into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.2). Cover and heat gently until the beryllium is completely dissolved. 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 Be.

4.7 Beryllium standard solution, 0,010 g/l Be

Transfer 5,0 ml of the beryllium stock solution (4.6) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,010 mg of Be.

4.8 Beryllium standard solution, 0,005 g/l Be

Transfer 50,0 ml of the beryllium standard solution (4.7) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,005 mg of Be.

4.9 Copper base solution, 5 g/l Cu

Weigh (5 ± 0,001) g of beryllium-free copper (Be ≤ 0,001 5 %) and transfer it into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.3) and, cautiously, 50 ml of nitric acid solution (4.5). Heat gently until the copper is dissolved and then bring to the boiling point until the nitrous fumes have been expelled. 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 5 mg of Cu.

4.10 Copper base solution, 1 g/l Cu

Transfer 50,0 ml of the copper base solution (4.9) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cu.

5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Atomic absorption spectrometer, fitted with a nitrous oxide/acetylene burner.

5.3 Beryllium 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. Add 10 ml of hydrochloric acid (4.2) and 10 ml of the nitric acid solution (4.5). Cover with a watch glass and heat gently until the test portion is completely dissolved, then heat at a temperature of approximately 90 °C until brown fumes have been expelled. Allow to cool. Wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

7.1.3 Beryllium mass fractions between 0,01 % and 0,10 %

Transfer 10 ml of the test portion solution (7.1.2) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

7.1.4 Beryllium mass fractions between 0,10 % and 0,5 %

Transfer 2,5 ml of the test portion solution (7.1.2) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

7.1.5 Beryllium mass fractions between 0,4 % and 3,0 %

Transfer 10 ml of the test portion solution (7.1.2) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Transfer 5 ml of this solution into a 200 ml one-mark volumetric flask. Add 5 ml of the 1 g/l copper solution (4.10), dilute to the mark with water and mix well.

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 beryllium 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 effects of alloying elements. If any 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 beryllium 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 concentration is a straight line.

7.4.1.2 Beryllium mass fractions between 0,01 % and 0,10 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of standard beryllium solutions (4.7) and copper base solutions (4.9) as shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for beryllium mass fractions between 0,01 % and 0,10 %

Beryllium standard solution volume (4.7)	Corresponding beryllium mass	Corresponding beryllium concentration after final dilution	Copper base solution volume (4.9)	Corresponding copper mass	Corresponding beryllium mass fraction of sample
ml	mg	mg/ml	ml	mg	%
0[a]	0	0	20	100	0
1	0,01	0,000 1	20	100	0,010
2,5	0,025	0,000 25	20	100	0,025
5	0,05	0,000 50	20	100	0,050
7,5	0,075	0,000 75	20	100	0,075
10	0,10	0,001 0	20	100	0,10

[a] Blank test on reagents for calibration curve.

7.4.1.3 Beryllium mass fractions between 0,10 % and 0,50 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of standard beryllium solutions (4.7) and copper base solutions (4.10) as shown in Table 2. Dilute to the mark with water and mix well.