SLOVENSKI STANDARD

SIST EN 14937-2:2006

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Baker in bakrove zlitine ï Določ evanje antimona ï 2. del: Metoda FAAS

Copper and copper alloys - Determination of antimony content - Part 2: FAAS method

Kupfer und Kupferlegierungen - Bestimmung des Antimongehaltes - Teil 2: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage de l'antimoine - Partie 2 : Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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English Version

Copper and copper alloys - Determination of antimony content - Part 2: FAAS method

Cuivre et alliages de cuivre - Dosage de l'antimoine - Partie 2 : Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des Antimongehaltes - Teil 2: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

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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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 14937-2:2006) 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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.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 14937-2, *Copper and copper alloys — Determination of antimony content* — Part 2*: FAAS method*

This is one of two parts of the standard for the determination of antimony content in copper and copper alloys. The other part is:

EN 14937-1, *Copper and copper alloys — Determination of antimony content — Part 1: Spectrophotometric 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 part of this European Standard specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the antimony content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having antimony mass fractions between 0,02 % and 2,0 %.

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 aqua regia followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 217,6 nm line emitted by an antimony hollow-cathode or electrodeless discharge 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 500 ml of nitric acid (4.3) in 500 ml of water.

4.5 Antimony stock solution, 1,000 g/l Sb

a) Weigh (1 ± 0,001) g of antimony (Sb > 99,90 %). Transfer it into a 250 ml beaker. Cover with a watch glass. Add 100 ml of hydrochloric acid (4.2) and 50 ml of nitric acid solution (4.4). Heat very gently until the antimony is completely dissolved. Cool to room temperature. Transfer it into a 1 000 ml one-mark volumetric flask, containing 200 ml of hydrochloric acid (4.2), dilute to the mark with water and mix well, or

b) Weigh 2,743 0 g of pure potassium antimony(III) oxide tartrate hemihydrate [$K(SbO)C_4H_4O_6 \cdot 1/2H_2O$] and dissolve it in water. 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 either solution contains 1,0 mg of Sb.

4.6 Antimony standard solution, 0,050 g/l Sb

Transfer 10,0 ml of the antimony stock solution (4.5) into a 200 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 0,050 mg of Sb.

4.7 Antimony standard solution, 0,200 g/l Sb

Transfer 20,0 ml of the antimony stock solution (4.5) 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 0,200 mg of Sb.

4.8 Copper base solution, 50 g/l Cu

Weigh 25,0 g of antimony-free copper (Cu ≥ 99,5 %) and transfer it into an 800 ml beaker. Add 125 ml of hydrochloric acid (4.2) and, cautiously, 250 ml of nitric acid solution (4.4). Cover with a watch glass and heat gently until the copper is completely dissolved, then continue heating to the boiling point. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water. Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 50 mg of Cu

4.9 Copper base solution, 10 g/l Cu

Transfer 50,0 ml of the copper base solution (4.8) into a 250 ml one-mark volumetric flask. Add 45 ml of hydrochloric acid (4.2) and 90 ml of nitric acid solution (4.4). Dilute to the mark with water and mix well.

1 ml of this solution contains 10 mg of Cu

5 Apparatus

5.1 Ordinary laboratory apparatus.

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

5.3 Antimony hollow-cathode or electrodeless discharge 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 Antimony mass fractions between 0,02 % and 0,10 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 5 ml of the hydrochloric acid (4.2) and 10 ml of the nitric acid solution (4.4). Cover and heat gently until the test portion is completely dissolved, then continue heating to the boiling point. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

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

7.1.3 Antimony mass fractions between 0,10 % and 2,0 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 5 ml of the hydrochloric acid (4.2) and 10 ml of the nitric acid solution (4.4). Cover and heat gently until the test portion is completely dissolved, then continue heating to the boiling point. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

Transfer quantitatively the dissolved test portion into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 20 ml of this solution into a 100 ml one-mark volumetric flask. 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 antimony 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 antimony concentration of the calibration solutions shall be adjusted to suit the sensitivity of the spectrometer used, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Antimony mass fractions between 0,02 % and 0,10 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of the antimony standard solution (4.6) and of the copper base solution (4.8) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for antimony mass fractions between 0,02 % and 0,10 %

Antimony standard solution volume (4.6) ml	Corresponding antimony mass mg	Corresponding antimony concentration after final dilution mg/ml	Copper base solution volume (4.8) ml	Corresponding copper mass g	Corresponding antimony mass fraction of sample %
0[a]	0	0	20	1,000	0
4	0,20	0,002 0	20	1,000	0,020
5	0,25	0,002 5	20	1,000	0,025
10	0,50	0,005 0	20	1,000	0,050
15	0,75	0,007 5	20	1,000	0,075
20	1,00	0,010	20	1,000	0,100

[a] Blank test on reagents for calibration curve.

7.4.1.3 Antimony mass fractions between 0,10 % and 2,0 %

Into each of a series of nine 100 ml one-mark volumetric flasks, introduce the volumes of the antimony standard solution (4.7) and of the copper base solution (4.9) shown in Table 2. Dilute to the mark with water and mix well.

Table 2 — Calibration for antimony mass fractions between 0,10 % and 2,0 %

Antimony standard solution volume (4.7) ml	Corresponding antimony mass mg	Corresponding antimony concentration after final dilution mg/ml	Copper base solution volume (4.9) ml	Corresponding copper mass g	Corresponding antimony mass fraction of sample %
0[a]	0	0	20	0,2	0
1	0,20	0,002 0	20	0,2	0,10
2	0,40	0,004 0	20	0,2	0,20
4	0,80	0,008 0	20	0,2	0,40
6	1,2	0,012	20	0,2	0,60
8	1,6	0,016	20	0,2	0,80
10	2,0	0,020	20	0,2	1,00
15	3,0	0,030	20	0,2	1,50
20	4,0	0,040	20	0,2	2,0

[a] Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the antimony hollow-cathode or electrodeless discharge lamp (5.3) into the atomic absorption spectrometer (5.2), switch on the current and allow to stabilize. Following the manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.