SLOVENSKI STANDARD
SIST EN 15915:2010

01-oktober-2010

Baker in bakrove zlitine - Določevanje srebra - Metoda z uporabo spektrometrije s plamensko atomsko absorpcijo (FAAS)

Copper and copper alloys - Determination of silver content - Flame atomic absorption spectrometric method (FAAS)

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

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

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Ta slovenski standard je istoveten z: **EN 15915:2010**

ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.120.30	Baker in bakrove zlitine	Copper and copper alloys

SIST EN 15915:2010 **en,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15915

July 2010

ICS 77.040.30; 77.120.30

English Version

Copper and copper alloys - Determination of silver content - Flame atomic absorption spectrometric method (FAAS)

Cuivre et alliages de cuivre - Détermination de l'argent - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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

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Foreword

This document (EN 15915:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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

EN 15915, *Copper and copper alloys — Determination of silver content — Flame atomic absorption spectrometric method (FAAS)*.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, 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 the United Kingdom.

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1 Scope

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

The methods are applicable to products having silver mass fractions between 0,01 % and 2,0 %.

a) Method A is applicable to copper and copper alloys having silver mass fractions between 0,01 % and 1,0 % and containing antimony or tin not greater than 0,005 0 % or silicon not greater than 0,010 %.

b) Method B is applicable to copper and copper alloys having silver mass fractions between 0,01 % and 2,0 % and antimony or tin greater than 0,005 % and silicon greater than 0,010 %.

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*

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

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3 Principle

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Dissolution of a test portion in an appropriate acid solution followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 328,1 nm line emitted by a silver hollow-cathode or electrodeless discharge lamp.

4 Reagents

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

WARNING — Special care shall be taken to strictly exclude chlorine and chloride ions from all operations, reagents, equipment and the laboratory air. For that reason all reagents indicated hereafter shall be freshly prepared and not stored after analysis.

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

4.2 Nitric acid solution, 1 + 1.

Add 500 ml of nitric acid (4.1) into 500 ml of water.

4.3 Boric acid, H_3BO_3 (40 g/l solution).

4.4 Hydrofluoric acid, HF, 48 % (ρ = 1,14 g/ml).

WARNING — Hydrofluoric acid is a hazardous substance. Care shall be taken and it shall be used under an efficient fume hood.

4.5 Fluoroboric-nitric acid mixture.

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In a 500 ml one-mark volumetric flask introduce:

— 150 ml of boric acid (4.3);

— 20 ml of hydrofluoric acid (4.4);

— 300 ml of nitric acid solution (4.2).

Dilute to the mark with water and mix well.

4.6 Silver stock solution, 1,0 g/l Ag.

Weigh (0,5 ± 0,001) g of silver (Ag ≥ 99,99 %) and transfer it into a 250 ml beaker. Add 50 ml of the nitric acid solution (4.2) and cover with a watch glass. Heat gently until the silver is dissolved and then boil or alternatively heat the solution in a boiling water bath until nitrous fumes have been expelled. Cool to room temperature, transfer the solution into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Ag.

4.7 Silver standard solution, 0,050 g/l Ag.

Transfer 10,0 ml of the silver stock solution (4.6) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,050 mg of Ag.

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4.8 Silver standard solution, 0,020 g/l Ag.

Transfer 5,0 ml of the silver stock solution (4.6) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

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1 ml of this solution contains 0,020 mg of Ag.

4.9 Copper base solution A, 10 g/l Cu.

For method A.

Weigh 5,0 g of pure copper (Ag ≤ 0,000 5 %) and transfer it into a 600 ml beaker. Add 100 ml of the nitric acid solution (4.2) and cover with a watch glass. Heat gently until the copper is completely dissolved, then boil or alternatively heat the solution in a boiling water bath 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.

4.10 Copper base solution B, 10 g/l Cu.

For method B.

Weigh 5,0 g of pure copper (Ag ≤ 0,000 5 %) and transfer it into a 600 ml plastic beaker. Add 75 ml of water, 10 ml of hydrofluoric acid (4.4) and by small fractions, 75 ml of nitric acid (4.1) and cover with a watch glass. Heat gently until the copper is completely dissolved, then heat the solution in a boiling water bath until nitrous fumes have been expelled. Cool to room temperature and transfer the solution quantitatively into a 500 ml plastic one-mark volumetric flask and add 75 ml of boric acid (4.3). Dilute to the mark with water and mix well.

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5 Apparatus

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

5.2 Silver 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 — Method A

7.1.1 General

Method A is applicable to products having silver mass fractions between 0,01 % and 1,0 % and containing antimony or tin not greater than 0,005 0 % or silicon not greater than 0,010 %.

7.1.2 Test portion

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Weigh (0,5 ± 0,001) g of the test sample.

7.1.3 Test portion solution

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7.1.3.1 General

Transfer the test portion (7.1.2) into a 200 ml conical flask and add 20 ml of the nitric acid solution (4.2). Warm gently until dissolution is complete, then boil or alternatively heat the solution in a boiling water bath until nitrous fumes have been expelled. Cool to room temperature.

7.1.3.2 Silver mass fractions below 0,2 %

Transfer the dissolved test portion (7.1.3.1) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

7.1.3.3 Silver mass fractions between 0,1 % and 1,0 %

Transfer the dissolved test portion (7.1.3.1) into a 500 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 substituting pure copper for the test portion (7.1.2).

7.3 Check test

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

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7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

The presence of copper in the calibration solutions compensates for chemical interaction effects of copper in the test 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 volumes of copper base solution A added (4.9) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Overcompensation may occur if the same volumes are added when the test samples are copper-based alloys where the percentage of copper is lower. In these cases the volumes of copper base solution shall be decreased to match the copper content of the test sample in solution.

NOTE The range of calibration solutions is appropriate for most current models of equipment of average performance. The range and operating conditions should be selected for optimum measurements by the particular equipment available.

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of silver standard solution (4.7), nitric acid solution (4.2) and copper base solution A (4.9) shown in Tables 1 or 2 depending on the expected silver content. Dilute to the mark with water and mix well.

Table 1 — Calibration for silver mass fractions between 0,01 % and 0,2 %

Silver standard solution volume (4.7) ml	**Corresponding silver mass** mg	**Corresponding silver concentration** mg/ml[b]	**Nitric acid solution** (4.2) ml	**Copper base solution A** (4.9) ml	**Corresponding copper mass** g	**Corresponding silver mass fraction of sample** %
0[a]	0	0	10	50	0,5	0
1	0,05	0,000 5	10	50	0,5	0,01
2	0,10	0,001 0	10	50	0,5	0,02
5	0,25	0,002 5	10	50	0,5	0,05
7	0,35	0,003 5	10	50	0,5	0,07
10	0,50	0,005 0	10	50	0,5	0,10
15	0,75	0,007 5	10	50	0,5	0,15
20	1,00	0,010 0	10	50	0,5	0,20

[a] Blank test on reagents for calibration curve.

[b] After final dilution.

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