SLOVENSKI STANDARD
SIST EN 15023-3:2010

01-oktober-2010

Nadomešča:
SIST-TS CEN/TS 15023-3:2007

Baker in bakrove zlitine - Določevanje niklja - 3. del: Metoda s plamensko atomsko absorpcijsko spektrometrijo (FAAS)

Copper and copper alloys - Determination of nickel content - Part 3: Flame atomic absorption spectrometry method (FAAS)

Kupfer und Kupferlegierungen - Bestimmung des Nickelgehaltes - Teil 3: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage du nickel - Partie 3: Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Ta slovenski standard je istoveten z: **EN 15023-3:2010**

ICS:

77.120.30 Baker in bakrove zlitine Copper and copper alloys

SIST EN 15023-3:2010 **en,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15023-3

July 2010

ICS 77.120.30

Supersedes CEN/TS 15023-3:2006

English Version

Copper and copper alloys - Determination of nickel content - Part 3: Flame atomic absorption spectrometric method (FAAS)

Cuivre et alliages de cuivre - Détermination du nickel - Partie 3: Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des Nickelgehaltes - Teil 3: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

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

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Foreword

This document (EN 15023-3:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.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.

This document supersedes CEN/TS 15023-3:2006.

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

CEN/TS 15023-3:2006, *Copper and copper alloys — Determination of nickel content — Part 3: Flame atomic absorption spectrometry method (FAAS)*.

In comparison with the first edition of CEN/TS 15023-3:2006, the following significant technical changes were made:

- Transformation into a European Standard;
- In 7.4.1.1, text added;
- Clause 9, Precision - completely revised.

This is one of the three parts of the standard for the determination of nickel content in copper and copper alloys. The other parts are:

- prEN 15023-1, *Copper and copper alloys — Determination of nickel content — Part 1: Spectrophotometric method*;
- prEN 15023-2, *Copper and copper alloys — Determination of nickel content — Part 2: Titrimetric method*.

Part 1 and Part 2 will be the subjects of future work.

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.

1 Scope

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

The method is applicable to products having a nickel mass fractions between 0,001 % and 6,0 %.

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*

3 Principle

Dissolution of a test portion in hydrochloric and nitric acid solution followed, after suitable dilution and the addition of lanthanum chloride to mask the effect of interfering ions, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 232,0 nm or the 352,4 nm line emitted by a nickel hollow-cathode lamp.

4 Reagents

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

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

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

4.3 Nitric acid solution, 1 + 1.

Add 500 ml of nitric acid (4.2) to 500 ml of water.

4.4 Lanthanum (III) chloride solution, 100 g/l.

Dissolve 50 g of lanthanum (III) chloride heptahydrate ($LaCl_3 \cdot 7H_2O$) in a 400 ml beaker with water and transfer the solution into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4.5 Nickel stock solution, 1,0 g/l Ni.

Weigh (1 ± 0,001) g of nickel (Ni ≥ 99,8 %) and transfer it into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.1) and 10 ml of the nitric acid solution (4.3). Cover with a watch glass and, if necessary, heat gently to assist dissolution. When dissolution is complete, cool to room temperature and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1,0 mg of Ni.

4.6 Nickel standard solution, 0,1 g/l Ni.

Transfer 20,0 ml of nickel stock solution (4.5) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,1 mg of Ni.

4.7 Nickel standard solution, 0,01 g/l Ni.

Transfer 5,0 ml of nickel stock solution (4.5) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,01 mg of Ni.

4.8 Copper base solution, 40 g/l Cu.

Transfer 20,0 g of nickel-free copper (Cu ≥ 99,95 %) into an 1 000 ml beaker. Add 100 ml of hydrochloric acid (4.1) and, cautiously, 200 ml of nitric acid solution (4.3). Cover with a watch glass and heat gently until the copper has been completely dissolved, then heat up to the boiling point until the 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.

4.9 Copper base solution, 2 g/l Cu.

Transfer 25,0 ml of copper base solution (4.8) into a 500 ml one-mark volumetric flask. Add 95 ml of hydrochloric acid (4.1) and 190 ml of nitric acid solution (4.3). Dilute to the mark with water and mix.

5 Apparatus

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

5.2 Nickel 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 General

Prepare test portion solutions in accordance with 7.1.2, 7.1.3 or 7.1.4 depending on the expected nickel content of the test sample.

7.1.2 Nickel mass fractions between 0,001 % and 0,012 5 %

Weigh (2 ± 0,001) g of the test sample.

Transfer the test portion into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.1) and 20 ml of the nitric acid solution (4.3). Cover with a watch glass and heat gently until the test portion is completely dissolved. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask. Add 10 ml of the lanthanum (III) chloride solution (4.4). Dilute to the mark with water and mix.

7.1.3 Nickel mass fractions between 0,01 % and 0,25 %

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

Transfer the test portion into a 250 ml beaker. Add 5 ml of hydrochloric acid (4.1) and 10 ml of the nitric acid solution (4.3). Cover with a watch glass and heat gently until the test portion is completely dissolved. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask. Add 10 ml of the lanthanum (III) chloride solution (4.4). Dilute to the mark with water and mix.

NOTE The validation exercise of this standard method showed that for nickel mass fractions between 0,2 % and 0,25 % results are better when the determination is carried out following 7.1.3.

7.1.4 Nickel mass fractions between 0,2 % and 6,0 %

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

Transfer the test portion into a 250 ml beaker. Add 5 ml of hydrochloric acid (4.1) and 10 ml of the nitric acid solution (4.3). Cover with a watch glass and heat gently until the test portion is completely dissolved. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 10,0 ml of this solution into a 200 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.1), 20 ml of the nitric acid solution (4.3) and 20 ml of the lanthanum (III) chloride solution (4.4). Dilute to the mark with water and mix.

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 reference material or a synthetic sample containing a known amount of nickel and of a 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 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 solutions added (4.8 and 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.

The nickel 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 Nickel mass fractions between 0,001 % and 0,012 5 %

Into each of a series of seven 100 ml one-mark volumetric flasks, introduce the volumes of nickel standard solution (4.7) and copper base solution (4.8) shown in Table 1. Add 10 ml of lanthanum (III) chloride solution (4.4). Dilute to the mark with water and mix.

Table 1 — Calibration for nickel mass fractions between 0,001 % and 0,012 5 %

Nickel standard solution volume (4.7)	Corresponding nickel mass	Corresponding nickel concentration after final dilution	Copper base solution volume (4.8)	Corresponding copper mass	Corresponding nickel mass fraction of sample
ml	mg	µg/ml	ml	g	%
0[a]	0	0	50	2	0
2	0,02	0,2	50	2	0,001 0
6	0,06	0,6	50	2	0,003 0
10	0,10	1,0	50	2	0,005 0
15	0,15	1,5	50	2	0,007 5
20	0,20	2,0	50	2	0,010 0
25	0,25	2,5	50	2	0,012 5

[a] Blank test on reagents for calibration curve.

7.4.1.3 Nickel mass fractions between 0,01 % and 0,25 %

Into each of a series of eight 100 ml one-mark volumetric flasks, introduce the volumes of nickel standard solution (4.6) and copper base solution (4.8) shown in Table 2. Add 10 ml of the lanthanum (III) chloride solution (4.4). Dilute to the mark with water and mix.