SLOVENSKI STANDARD
SIST EN 15025:2010

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Nadomešča:
SIST-TS CEN/TS 15025:2007

Baker in bakrove zlitine - Določevanje magnezija - Metoda s plamensko atomsko absorpcijsko spektrometrijo (FAAS)

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

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

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

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English Version

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

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

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

Foreword

This document (EN 15025:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.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 15025:2006.

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 15025:2006, *Copper and copper alloys — Determination of magnesium content — Flame atomic absorption spectrometry method (FAAS)*.

In comparison with the first edition of CEN/TS 15025:2006, the following significant technical changes were made:

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

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 magnesium content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having magnesium mass fractions between 0,001 % and 0,20 %.

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 a hydrochloric-nitric acid mixture followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Determination of the magnesium content by measuring the absorption of the 285,2 nm line emitted by a magnesium 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 100 ml of nitric acid (4.2) to 100 ml of water.

4.4 Magnesium stock solution, 0,5 g/l Mg.

Weigh (0,5 ± 0,001) g of magnesium (Mg ≥ 99,9 %) and transfer it into a 250 ml beaker. Add 20 ml of the nitric acid solution (4.3) in small amounts. Cover with a watch glass and heat gently until the magnesium is completely dissolved. Boil the solution until nitrous fumes have been expelled. 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 well.

1 ml of this solution contains 0,5 mg of Mg.

4.5 Magnesium standard solution, 0,01 g/l Mg.

Transfer 5 ml of the magnesium stock solution (4.4) into a 250 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 0,01 mg of Mg.

4.6 Magnesium standard solution, 0,000 5 g/l Mg.

Transfer 5 ml of the magnesium standard 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 0,000 5 mg of Mg.

4.7 Lanthanum (III) chloride solution, 200 g/l.

Dissolve 100 g of lanthanum chloride heptahydrate ($LaCl_3 \cdot 7H_2O$) in a 1 000 ml beaker with approximately 400 ml of water. Transfer the solution into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

4.8 Copper base solution, 10 g/l Cu.

Weigh (10 ± 0,01) g of electrolytic copper and transfer it into a 1 000 ml beaker. Add 100 ml of hydrochloric acid (4.1) and, in small amounts, 100 ml of the nitric acid solution (4.3). Cover with a watch glass and heat gently until the copper is dissolved, then boil until the nitrous fumes have been expelled. Cool to room temperature and transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

5 Apparatus

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

5.2 Magnesium 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 (4.1) 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, then heat at a temperature of approximately 90 °C until nitrous fumes have been expelled. Cool to room temperature. Wash the cover and the sides of the beaker with water. Transfer the solution to a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Select an aliquot portion from the test portion solution according to the expected magnesium mass fraction as indicated in Table 1.

Table 1 — Aliquot solution

Magnesium (mass fraction)	Aliquot of the test portion solution (7.1.2)	Lanthanum (III) chloride solution volume (4.7)	Final volume of diluted solution
%	ml	ml	ml
0,001 to 0,05	10	10	100
0,05 to 0,2	5	10	100

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 reference material or a synthetic sample containing a known amount of magnesium and of a composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Preparation 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 solution added (4.8) 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 magnesium 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 Magnesium mass fractions between 0,001 % and 0,20 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of magnesium standard solution (4.5 or 4.6) and of copper base solution (4.8) shown in Tables 2 or 3, depending on the expected magnesium content, followed by 10 ml of lanthanum (III) chloride solution (4.7). Dilute to the mark with water and mix well.

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

Table 2 — Calibration for magnesium mass fractions between 0,001 % and 0,05 %

Magnesium standard solution volume (4.6)	Magnesium standard solution volume (4.5)	Corresponding magnesium mass	Corresponding magnesium concentration after final dilution	Copper base solution volume (4.8)	Corresponding copper mass	Corresponding magnesium mass fraction of sample
ml	ml	mg	mg/ml	ml	g	%
0[a]	—	0	0	10	0,1	0
2	—	0,001	0,000 01	10	0,1	0,001
4	—	0,002	0,000 02	10	0,1	0,002
10	—	0,005	0,000 05	10	0,1	0,005
20	—	0,01	0,000 1	10	0,1	0,010
—	2	0,02	0,000 2	10	0,1	0,020
—	3	0,03	0,000 3	10	0,1	0,030
—	4	0,04	0,000 4	10	0,1	0,040
—	5	0,05	0,000 5	10	0,1	0,050

[a] Blank test on reagents for calibration curve.

Table 3 — Calibration for magnesium mass fractions between 0,05 % and 0,20 %

Magnesium standard solution volume (4.5)	Corresponding magnesium mass	Corresponding magnesium concentration after final dilution	Copper base solution volume (4.8)	Corresponding copper mass	Corresponding magnesium mass fraction of sample
ml	mg	mg/ml	ml	g	%
0[a]	0	0	5	0,050	0
2,5	0,025	0,000 25	5	0,050	0,05
5,0	0,050	0,000 50	5	0,050	0,10
7,5	0,075	0,000 75	5	0,050	0,15
10,0	0,1	0,001	5	0,050	0,20

[a] Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the magnesium hollow-cathode lamp (5.2) into the atomic absorption spectrometer (5.1), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 285,2 nm to minimum absorbance. 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.