



SLOVENSKI STANDARD
SIST-TS CEN/TS 15025:2007

01-februar-2007

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Copper and copper alloys - Determination of magnesium content - Flame atomic absorption spectrometry 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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Ta slovenski standard je istoveten z: CEN/TS 15025:2006

ICS:

77.120.30 Baker in bakrove zlitine Copper and copper alloys

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ICS 77.120.30

English Version

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

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flamme (SAAF)

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

This Technical Specification (CEN/TS) was approved by CEN on 12 September 2006 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (CEN/TS 15025:2006) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

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

CEN/TS 15025, Copper and copper alloys — Determination of magnesium content — Flame atomic absorption spectrometry method (FAAS)

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: 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 the United Kingdom.

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1 Scope

This Technical Specification 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 Technical Specification. 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 and materials

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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 ($\rho = 1,19$ g/ml)

4.3 Nitric acid, HNO₃ ($\rho = 1,40$ g/ml)

4.4 Nitric acid solution, 1 + 1

Dilute 100 ml of nitric acid (4.3) in 100 ml of water.

4.5 Magnesium stock solution, 0,5 g/l Mg

Weigh ($0,5 \pm 0,001$) g of magnesium ($Mg \geq 99,9$ %) and transfer it into a 250 ml beaker. Add 20 ml of the nitric acid solution (4.4) 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, 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.

4.6 Magnesium standard solution, 0,01 g/l Mg

Using a calibrated pipette, transfer 5 ml of the magnesium stock solution (4.5) 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.

4.7 Magnesium standard solution, 0,000 5 g/l Mg

Using a calibrated pipette, transfer 5 ml of the magnesium standard solution (4.6) 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.

4.8 Lanthanum (III) chloride solution, 200 g/l

Dissolve 200 g of lanthanum chloride heptahydrate ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) in a 2 000 ml beaker with approximately 800 ml of water. Transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

4.9 Copper base solution, 50 g/l Cu

Weigh ($50 \pm 0,01$) g of electrolytic copper and transfer it into a 2 000 ml beaker. Add 500 ml of hydrochloric acid (4.2) and, in small amounts, 250 ml of the nitric acid solution (4.4). 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.

4.10 Copper base solution, 10 g/l Cu

Transfer 50 ml of the copper base solution (4.9) into a 250 ml one-mark volumetric flask, add 15 ml of the nitric acid solution (4.4). Dilute to the mark with water and mix well.

1 ml of this solution contains 0,01 g of Cu.

5 Apparatus

5.1 Ordinary laboratory apparatus

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

5.3 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 0,3 mm.

7 Procedure

7.1 Preparation of the test portion solution

7.1.1 Test portion

Weigh ($1 \pm 0,001$) g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker (5.2). Add 10 ml of hydrochloric acid (4.2) and 10 ml of the nitric acid solution (4.4). 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. Wash the cover and the sides of the beaker with water. Cool to room temperature. 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) ml	Lanthanum chloride(III) solution volume (4.8) ml	Final volume of diluted solution 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 standard 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 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 (4.6 or 4.7) and of copper base solution (4.10) shown in Tables 2 or 3, depending on the expected magnesium content, followed by 10 ml of lanthanum (III) chloride solution (4.8). 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		Corresponding magnesium mass	Corresponding magnesium concentration after final dilution	Copper base solution volume	Corresponding copper mass	Corresponding magnesium mass fraction of sample
(4.7)	(4.6)					
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	Corresponding magnesium mass	Corresponding magnesium concentration after final dilution	Copper base solution volume	Corresponding copper mass	Corresponding magnesium mass fraction of sample
(4.6)			(4.10)		
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	0,050	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.