



SLOVENSKI STANDARD
SIST EN 15022-3:2006

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

Kupfer und Kupferlegierungen - Bestimmung des Zinngehaltes - Teil 3: Niedriger Zinngehalt - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 3 : Etain en faible teneur - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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Ta slovenski standard je istoveten z: EN 15022-3:2006

ICS:

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ICS 77.120.30

English Version

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

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 3 :
Etain en faible teneur - Méthode par spectrométrie
d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des
Zinngehaltes - Teil 3: Niedriger Zinngehalt -
Flammenatomabsorptionsspektrometrisches Verfahren
(FAAS)

This European Standard was approved by CEN on 14 August 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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.

CEN members are the national standards bodies of 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.



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Foreword

This European Standard (EN 15022-3:2006) 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 April 2007, and conflicting national standards shall be withdrawn at the latest by April 2007.

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 15022-3, *Copper and copper alloys — Determination of tin content — Part 3: Low tin content — Flame atomic absorption spectrometry method (FAAS)*

This is one of four parts of the standard/Technical Specification for the determination of tin content in copper and copper alloys. The other parts are:

prEN 15022-1, *Copper and copper alloys — Determination of tin content — Part 1: Titrimetric method*

prEN 15022-2, *Copper and copper alloys — Determination of tin content — Part 2: Spectrometric method*

prEN 15022-4, *Copper and copper alloys - Determination of tin content - Part 4: Medium tin 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 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.

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1 Scope

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

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

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. 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 acid and hydrogen peroxide. Extraction of tin into the organic phase with a solution of trioctylphosphine oxide in methylisobutylketone followed by aspiration of the organic solution into a nitrous oxide/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 286,3 nm line emitted by a tin hollow-cathode lamp.

4 Reagents and materials

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4.1 General

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

4.2 Ethanol, C₂H₅OH

4.3 4-methylpentan-2-one (methylisobutylketone) (M.I.B.K.), CH₃COCH₂CH(CH₃)₂

WARNING — 4-methylpentan-2-one is a volatile, flammable organic material. Normal safety precautions should be taken.

4.4 M.I.B.K. alcoholic solution

Mix 92 parts of M.I.B.K. solution (4.3) with 8 parts of ethanol (4.2).

4.5 Hydrochloric acid, HCl ($\rho = 1,19$ g/ml)

4.6 Hydrochloric acid solution, 1 + 1

Dilute 500 ml of hydrochloric acid (4.5) in 500 ml of water.

4.7 Hydrogen peroxide, H_2O_2 30 % (mass fraction) solution, free from tin base stabilizers

4.8 Trioctylphosphine oxide (T.O.P.O.), $[\text{CH}_3(\text{CH}_2)_7]_3\text{PO}$ 25 g/l solution in 4-methylpentan-2-one

In a 200 ml one-mark volumetric flask, dissolve 5 g of trioctylphosphine oxide $[\text{CH}_3(\text{CH}_2)_7]_3\text{PO}$ in 4-methylpentan-2-one (4.3). Dilute to the mark with 4-methylpentan-2-one and mix well.

4.9 T.O.P.O. alcoholic solution

Mix 92 parts of T.O.P.O. solution (4.8) with 8 parts of ethanol (4.2).

4.10 Tin stock solution, 0,5 g/l Sn

Weigh $(0,5 \pm 0,001)$ g of tin ($\text{Sn} \geq 99\%$) and transfer it into a 500 ml narrow-necked conical flask. Add 100 ml of hydrochloric acid (4.5) and cover with a watch glass. When dissolution is complete, add 100 ml of water and several drops of hydrogen peroxide solution (4.7). Cool and transfer this 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 Sn.

4.11 Tin standard solution, 0,05 g/l Sn

Using a calibrated pipette, transfer 10 ml of the tin stock solution (4.10) into a 100 ml one-mark volumetric flask. Add 9 ml of hydrochloric acid (4.5) and dilute to about 90 ml with water. Cool, dilute to the mark with water and mix well.

1 ml of this solution contains 0,05 mg of Sn.

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5 Apparatus

5.1 Ordinary laboratory apparatus

5.2 Atomic absorption spectrometer, fitted with a nitrous oxide/acetylene burner.

5.3 Tin 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 ($5 \pm 0,001$) g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker and cover. Add 30 ml of hydrochloric acid (4.5) and in small portions, 40 ml of the hydrogen peroxide solution (4.7), cooling if necessary. When the test portion is completely dissolved, heat gently to decompose the hydrogen peroxide. Cool and transfer into a 250 ml separatory funnel, rinsing the beaker with about 10 ml of water.

7.1.3 Extraction of tin

Add 20 ml of the T.O.P.O. solution (4.8) to the separatory funnel containing the test portion solution (7.1.2), close with a stopper and shake for 1 min. Allow to stand for about 5 min until the phases have separated, then discard the aqueous phase. Wash the organic phase twice with 20 ml of the hydrochloric acid solution (4.6), shaking gently for several seconds to eliminate most of the copper. The remaining trace of copper will not interfere. Transfer the washed organic phase into a 25 ml one-mark volumetric flask containing 2 ml ethanol (4.2). Rinse the separatory funnel with several ml of T.O.P.O. solution (4.8) and add to the volumetric flask. Dilute to the mark with T.O.P.O. solution (4.8) 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 tin 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

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, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Calibration solutions

Into each of a series of eight 250 ml separatory funnels, transfer 30 ml of hydrochloric acid (4.5), 50 ml of water and the volumes of tin stock solution (4.10) and tin standard solution (4.11) shown in Table 1.

Table 1 — Calibration for tin mass fractions between 0,001 % and 0,60 %

Tin stock or standard solution volume		Corresponding tin mass	Corresponding tin concentration after final dilution	Corresponding tin mass fraction (see 7.5.2)	
(4.11)	(4.10)			Direct test portion	Diluted 1:10 test portion
ml	ml	mg	mg/ml	%	%
0 ^a	—	0	0	0	0
1	—	0,050	0,002	0,001	—
3	—	0,15	0,006	0,003	0,03
6	—	0,30	0,012	0,006	0,06
10	—	0,50	0,020	0,010	0,10
—	2	1,0	0,040	0,02	0,20
—	4	2,0	0,080	0,04	0,40
—	6	3,0	0,120	—	0,60

^a Blank test on reagents for calibration curve.

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Add 20 ml of the T.O.P.O. solution (4.8) to each separatory funnel, close with a stopper and shake for 1 min. Allow to stand for about 5 min until the phases have separated, then discard the aqueous phase. Transfer each organic phase into eight 25 ml one-mark volumetric flasks each containing 2 ml of ethanol (4.2). Rinse the separatory funnel with several ml of T.O.P.O. solution (4.8) and add to the volumetric flasks. Dilute to the mark with T.O.P.O. solution (4.8) and mix well.

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7.4.2 Adjustment of the atomic absorption spectrometer

Fit the tin hollow-cathode lamp (5.3) into the atomic absorption spectrometer (5.2), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 286,3 nm to maximum energy. 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. Also take into account that the flame should be slightly reducing with a red zone height of approximately 7 mm to 12 mm when aspirating the solution 4.9.

NOTE The wavelength noted, 286,3 nm, was chosen because of its stability, linearity of calibration curve, sensitivity and lack of interference. Similar sensitivity can be obtained by using 235,5 nm wavelength. Use of other lines is not advised.

7.4.3 Spectrometric measurement of the calibration solutions

Aspirate the series of calibration solutions (7.4.1) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray M.I.B.K. alcoholic solution (4.4) through the burner after each measurement.

7.4.4 Calibration curve

Establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or an off-line computer for regression calculations or prepare a graphical representation.