



SLOVENSKI STANDARD
SIST-TS CEN/TS 15022-4:2007

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Copper and copper alloys - Determination of tin content - Part 4: Medium tin content -
Flame atomic absorption spectrometry method (FAAS)

Kupfer und Kupferlegierungen - Bestimmung des Zinngehaltes - Teil 4: Mittlerer
Zinngehalt - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 4 : Etain en moyenne teneur -
Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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Ta slovenski standard je istoveten z: CEN/TS 15022-4:2006

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 tin content - Part 4:
Medium tin content - Flame atomic absorption spectrometry
method (FAAS)**

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 4 :
Etain en moyenne teneur - Méthode par spectrométrie
d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des
Zinngehaltes - Teil 4: Mittlerer Zinngehalt -
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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Foreword

This document (CEN/TS 15022-4:2006) 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 15022-4, *Copper and copper alloys — Determination of tin content — Part 4: Medium 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*

EN 15022-3, *Copper and copper alloys — Determination of tin content — Part 3: Low 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 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 the tin content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having medium tin mass fractions between 0,2 % and 3 %.

2 Normative references

The following referenced documents are indispensable for the application of this European 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 hydrochloric acid and hydrogen peroxide followed, after suitable dilution, by aspiration 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 discharge lamp.

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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 Hydrochloric acid solution, 7 + 3

Dilute 700 ml of hydrochloric acid (4.2) in 300 ml of water.

4.4 Hydrogen peroxide, H₂O₂ 30 % (mass fraction) solution, free from tin base stabilizers

Hydrogen peroxide may be stabilized by products containing some tin. It is therefore necessary to use exactly the same volume of hydrogen peroxide for the dissolution of the test sample as for the preparation of the calibration solution.

4.5 Tin stock solution, 1 g/l Sn

Weigh ($1 \pm 0,001$) g of tin ($\text{Sn} \geq 99$ %) and transfer it into a 250 ml beaker. Dissolve it in 100 ml hydrochloric acid (4.2) and several drops of hydrogen peroxide (4.4) and cover with a watch glass. Heat gently until the metal is dissolved. 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 1 mg of Sn.

4.6 Copper base solution, 20 g/l Cu

Weigh 10,0 g of electrolytic copper ($\text{Cu} \geq 99,95\%$) into a 600 ml beaker. Add 250 ml of the hydrochloric acid solution (4.3) and cover with a watch glass. Cool and add successively 5 ml hydrogen peroxide portions (4.4) until dissolution is complete, waiting after each addition until all effervescence ceases. Heat to eliminate the excess hydrogen peroxide. Cool and transfer this solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

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.

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7 Procedure

7.1 Preparation of the test portion solution

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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 and cover with a watch glass. Add 25 ml of hydrochloric acid solution (4.3), cool and add 5 ml of hydrogen peroxide (4.4). Cool until the violent reaction has ceased and add, if necessary, 2 ml of hydrogen peroxide to continue the dissolution. Repeat this addition several times, as necessary to complete the dissolution of the test portion. Heat to eliminate the excess of hydrogen peroxide and cool. Transfer the dissolved test portion into a 100 ml one-mark volumetric flask. Add 3,5 ml of hydrochloric acid (4.2). 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.

7.3 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 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 and chloride 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 portion solution.

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

7.4.1.2 Tin mass fractions between 0,2 % and 3,0 %

Into each of a series of five 100 ml one-mark volumetric flasks, introduce the volumes of the tin stock solution (4.5), the copper base solution (4.6) and hydrochloric acid (4.2) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for tin mass fractions between 0,2 % and 3,0 %

Tin stock solution volume (4.5) ml	Corresponding tin mass mg	Corresponding tin concentration after final dilution mg/ml	Copper base solution volume (4.6) ml	Corresponding copper mass g	Hydrochloric acid volume (4.2) ml	Corresponding tin mass fraction of sample %
0 ^a	0	0	50	1	3,5	0
2	2	0,02	50	1	3,3	0,20
5	5	0,05	50	1	3,0	0,50
10	10	0,10	50	1	2,5	1,00
15	15	0,15	50	1	2,0	1,50
20	20	0,20	50	1	1,5	2,00
25	25	0,25	50	1	1,0	2,50
30	30	0,30	50	1	0,5	3,00

^a Blank test on reagents for calibration curve.

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 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.

NOTE The wavelength 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 the 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 (see 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 water through the burner after each measurement, see NOTE.

NOTE For certain types of apparatus, instead of water it is preferable to use a solution containing the attack reagents, in the same concentrations as in the test portion solutions.

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.

7.5 Determination

7.5.1 General

The analyses shall be carried out independently, in duplicate.

7.5.2 Preliminary spectrometric measurement

Carry out a preliminary measurement on the test portion solution (7.1.2) following the same procedure specified in 7.4.2 and 7.4.3 at the same time as the spectrometric measurements are carried out on the calibration solutions (see 7.4.1). Estimate the preliminary analyte amount by using the calibration curve (7.4.4).

7.5.3 Spectrometric measurements (standards.iteh.ai)

7.5.3.1 Use of the calibration curve SIST-TS CEN/TS 15022-4:2007 <https://standards.iteh.ai/catalog/standards/sist/aace5cb2-8f99-4a76-b056-8924f1b6753/cen-ts-15022-4:2007>

Repeat the measurements and determine the concentration directly using the calibration curve.

7.5.3.2 Use of bracketing method

Carry out a second measurement on the test portion solution (7.1.2) following the procedure specified in 7.4.3, by bracketing between two new calibration solutions with composition similar to that of the calibration solution (see 7.4.1), but having tin contents slightly higher and slightly lower ($\pm 10\%$) than the estimated tin concentration of the test portion solution.

To prepare these calibration solutions, follow the procedure specified in 7.4.1 using, however, suitable quantities of tin stock solutions (4.5).

8 Expression of results

8.1 Use of calibration curve

Calculate the tin mass fraction, in percent (%), as follows:

$$\text{Tin mass fraction} = \frac{A_1}{B} \times V_f \times 100 \quad (1)$$

where

A_1 is the tin concentration from the calibration curve, in milligram per millilitre (mg/ml);

B is the sample mass represented in the test portion, in milligram (mg);

V_f is the volume of the test portion solution (7.1.2), in millilitre (ml).