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SIST-TS CEN/TS 15022-2:2010
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Copper and copper alloys - Determination of tin content - Part 2: Spectrophotometric method

Kupfer und Kupferlegierungen - Bestimmung des Zinngehaltes - Teil 2: Spektrophotometrisches Verfahren

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 2 : Méthode spectrophotométrique

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ICS:

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**Copper and copper alloys - Determination of tin content - Part 2:
Spectrophotometric method**

Cuivre et alliages de cuivre - Dosage de l'étain - Partie 2 :
Méthode spectrophotométrique

Kupfer und Kupferlegierungen - Bestimmung des
Zinngehaltes - Teil 2: Spektrophotometrisches Verfahren

This Technical Specification (CEN/TS) was approved by CEN on 15 September 2009 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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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (CEN/TS 15022-2:2009) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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

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* (Part 1 will be the subject of future work);

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

CEN/TS 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 announce this Technical Specification: Austria, Belgium, Bulgaria, 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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CEN/TS 15022-2:2009 (E)**1 Scope**

This part of this Technical Specification specifies the spectrophotometric method 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 tin mass fractions between 0,005 % and 0,5 %.

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

Extraction into methyl isobutyl ketone and spectrophotometric determination of the yellow tin-quercetin complex at a wavelength of 440 nm.

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

4.2 Methyl isobutyl ketone.

4.3 Ammonia solution, NH₄OH ($\rho = 0,91$ g/ml).

4.4 Hydrogen peroxide solution, H₂O₂, 30 % (mass fraction).

4.5 Hydrochloric acid solution, diluted 1 + 1.

Add 50 ml of hydrochloric acid (4.1) to 50 ml of water.

4.6 Sulphuric acid, H₂SO₄ ($\rho = 1,84$ g/ml).

4.7 Sulphuric acid solution, diluted 1 + 19.

Add 50 ml of sulphuric acid (4.6) to 950 ml of water.

4.8 Thiourea solution, NH₂CSNH₂ (60 g/l).

Dissolve 15 g of thiourea in water and dilute to 250 ml.

4.9 Ascorbic acid solution, $C_6H_8O_6$ (20 g/l).

Dissolve 1 g of ascorbic acid in water and dilute to 50 ml.

Use a freshly prepared solution.

4.10 Quercetin acid ethanol solution, (1 g/l).

Dissolve 500 mg of quercetin in 300 ml of 95 % (volume fraction) ethanol in a 500 ml one-mark volumetric flask. The dissolution takes some hours. Add 25 ml of the hydrochloric acid (4.1), dilute to the mark with water and mix well. Filter off any residue.

4.11 Tin stock solution, 0,5 g/l Sn.

Weigh $(0,5 \pm 0,001)$ g of pure tin ($Sn \geq 99,99\%$) and transfer it into a 400 ml beaker. Add 100 ml of hydrochloric acid (4.1) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling, 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 Sn.

4.12 Tin standard solution, 0,050 g/l Sn.

Transfer 10,0 ml of the tin stock solution (4.11) to a 100 ml one-mark volumetric flask. Add 20 ml of the hydrochloric acid (4.1). Dilute to the mark with water and mix well.

1 ml of this solution contains 50 μ g of Sn.

4.13 Copper base solution, 5 g/l

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Dissolve 5,0 g of electrolytic tin-free copper in 100 ml of the hydrochloric acid (4.1). Add 5 ml portions of the hydrogen peroxide solution (4.4) until the copper is completely dissolved then add 200 ml of water and boil to destroy the excess of the hydrogen peroxide. Cool and transfer to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

5 Apparatus

5.1 Spectrophotometer, with 20 mm cells.

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

7.1.1 Test portion

Weigh to the nearest 0,001 g, according to the expected tin mass fraction of the test sample, a test portion according to Table 1.

Table 1 — Test portion mass

Expected tin mass fraction of test sample %	Mass of test portion ($\pm 0,001$ g) g	Predilution	
		aliquot volume taken ml	diluted to ml
0,005 to 0,010	2	without predilution	
0,01 to 0,02	1		
0,02 to 0,04	1	100	200
0,04 to 0,08	1	50	200
0,08 to 0,16	1	25	200
0,16 to 0,30	0,5	25	200
0,30 to 0,50	0,4	20	200

7.1.2 Test portion solution

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Transfer the test portion to a 250 ml conical flask.

Under cold running water, dissolve the test portion in 20 ml of the hydrochloric acid solution (4.5) and 10 ml of the hydrogen peroxide solution (4.4), in small portions. It is necessary to dissolve slowly to avoid loss of tin chloride. Complete the dissolution by heating gently. Add 10 ml of water and boil to destroy the excess of hydrogen peroxide. Transfer the test portion solution to a 200 ml one-mark volumetric flask.

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 (7.1.1). Correct the result obtained from the determination in accordance with the result from the blank test.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of 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 7.1 and 7.5.

7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

The tin concentration of the calibration solutions was adjusted to meet the sensitivity of common apparatus, so that the curve of integrated absorbance as a function of concentration is a straight line. If the calibration curve is not a straight line, use a 10 mm cell.

7.4.1.2 Tin mass fraction between 0,005 % and 0,5 %

Into each of a series of seven 200 ml one-mark volumetric flasks introduce 50 ml of the copper base solution (4.13) and 10 ml of water. Add of the tin standard solution (4.12) 0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml, 4,0 ml and 5,0 ml, equivalent to 0 µg, 25 µg, 50 µg, 100 µg, 150 µg, 200 µg and 250 µg of tin. Proceed as specified in 7.5.2, 7.5.3 and 7.5.4.

7.4.2 Spectrophotometric measurements of the calibration solutions

Measure the absorbance of the solutions using the spectrophotometer (5.1) fitted with a 20 mm cell, against methyl-isobutyl ketone at 440 nm.

7.4.3 Calibration curve

Establish the calibration curve using measured absorbances, after correcting the results for the zero solution and corresponding analyte amounts. Use appropriate spectrophotometer software or off-line computer for regression calibration or prepare a graphical representation.

7.5 Determination

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7.5.1 The analyses shall be carried out independently, in duplicate. If, according to the Table 1, no dilution is to be made, proceed as directed in 7.5.2, 7.5.3 and 7.5.4. If a predilution is to be made, dilute to 200 ml with water and mix well. Transfer the prescribed aliquot portion to a 200 ml one-mark volumetric flask.

7.5.2 Neutralize with the ammonia solution (4.3) just to the point where a precipitate appears and remains. Dissolve the precipitate with the hydrochloric acid (4.1) added drop by drop. Add 20 ml of water and 30 ml of the hydrochloric acid. Allow to cool, dilute to the mark with water and mix well.

7.5.3 Introduce successively into a 125 ml separating funnel:

- a) 25 ml of the thiourea solution (4.8);
- b) 5 ml of the ascorbic acid solution (4.9);
- c) 10,0 ml of the quercetin acid ethanol solution (4.10) and mix;
- d) 10,0 ml of the test portion solution while mixing;
- e) 20,0 ml of the methyl isobutyl ketone (4.2).

7.5.4 Shake for 1 min and allow the layers to separate for 3 min. Discard the aqueous layer. Add 5 ml of the sulphuric acid solution (4.7), without mixing. Drain off the acid phase and add 20 ml of the sulphuric acid solution. Shake for 30 s. During shaking avoid too close an intermixture of the phases; this will prolong the separation time. After 3 min, drain off and discard the acid phase and a little of the organic phase. Transfer the remainder of the organic phase through a small, dry, rapid filter paper into a dry, stoppered flask. After 10 min, measure the absorbance of the solution using a spectrophotometer (5.1), fitted with a 20 mm cell, against methyl isobutyl ketone at 440 nm.