



SLOVENSKI STANDARD

SIST EN 15616:2013

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Nadomešča:

SIST-TS CEN/TS 15616:2009

Baker in bakrove zlitine - Določevanje kadmija - Metoda z uporabo spektrometrije s plamensko atomsko absorpcijo (FAAS)

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

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

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

Ta slovenski standard je istoveten z: EN 15616:2012

ICS:

77.120.30 Baker in bakrove zlitine Copper and copper alloys

SIST EN 15616:2013

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EUROPEAN STANDARD

EN 15616

NORME EUROPÉENNE

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November 2012

ICS 77.120.30

Supersedes CEN/TS 15616:2009

English Version

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

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

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

This European Standard was approved by CEN on 20 October 2012.

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 CEN-CENELEC Management Centre 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 CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 15616:2012) 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 May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

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 15616:2009.

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

CEN/TS 15616:2009, *Copper and copper alloys — Determination of cadmium content — Flame atomic absorption spectrometric method (FAAS)*

In comparison with CEN/TS 15616:2009, the following significant technical changes were made:

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- a) Conversion into a European Standard;
 - b) Scope (Clause 1) revised;
 - c) Principle (Clause 3) completely revised, dissolution process changed;
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 - d) Precision (Clause 9) completely revised and the results of the precision test included.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

EN 15616:2012 (E)**1 Scope**

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

The method is applicable to products having cadmium mass fractions between 0,000 5 % and 0,1 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 nitric acid solution followed, after suitable dilution, by aspiration of the test solution into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 228,8 nm line emitted by a cadmium hollow-cathode lamp.

4 Reagents

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

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

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

4.3 Nitric acid solution, 1 + 1

Add 500 ml of nitric acid (4.2) to 500 ml of water.

4.4 Cadmium stock solution, 1,0 g/l Cd

Weigh ($1 \pm 0,001$) g of cadmium ($\text{Cd} \geq 99,8$ %) and transfer it into a 250 ml beaker. Add 10 ml of nitric acid (4.2). Cover with a watch glass and, if necessary, heat gently to assist dissolution. When dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1,0 mg of Cd.

4.5 Cadmium standard solution, 0,005 g/l Cd

Transfer 2,5 ml of cadmium stock solution (4.4) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,005 mg of Cd.

4.6 Copper base solution, 50 g/l Cu

Transfer 25,0 g of cadmium-free copper ($\text{Cu} \geq 99,95 \%$) into a 1 000 ml beaker. Add 250 ml of hydrochloric acid (4.1) and, cautiously, 125 ml of nitric acid (4.2). Cover with a watch glass and heat gently until the copper has been completely dissolved, then heat up to the boiling point until the nitrous fumes have been expelled. Allow to cool and transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

4.7 Copper base solution, 5 g/l Cu

Transfer 50,0 ml of copper base solution (4.6) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

5 Apparatus

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

5.2 Cadmium 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

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7.1 Preparation of the test portion solution

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7.1.1 General <https://standards.iteh.ai/catalog/standards/sist/421a9abc-d89b-481a-8286-6bbd61ecf90/sist-en-15616-2013>

Prepare test portion solutions in accordance with 7.1.2 or 7.1.3 depending on the expected cadmium content of the test sample.

7.1.2 Cadmium mass fractions between 0,000 5 % and 0,010 %

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

Transfer the test portion 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 and then bring to boiling point until the nitrous fumes have been expelled. Cool to room temperature, and wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

NOTE The validation test of this method showed that for cadmium mass fractions until and including 0,01 %, the results are better when the determination is carried out following 7.1.2.

7.1.3 Cadmium mass fractions between 0,01 % and 0,10 %

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

Transfer the test portion 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 and then bring to the boiling point until the nitrous fumes have been expelled. Cool to room temperature, and wash the beaker cover and the sides of the beaker with water.

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Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Transfer 5,0 ml of this solution into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

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 reference material or a synthetic sample containing a known amount of cadmium 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, 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 portion 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 (4.6 or 4.7) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Over-compensation 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 cadmium 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 Cadmium mass fractions between 0,000 5 % and 0,010 %

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

Table 1 — Calibration for cadmium mass fractions between 0,000 5 % and 0,010 %

Cadmium standard solution volume (4.5) ml	Corresponding cadmium mass mg	Corresponding cadmium concentration after final dilution µg/ml	Copper base solution volume (4.6) ml	Corresponding copper mass g	Corresponding cadmium mass fraction of sample %
0 ^a	0	0	20	1	0
1	0,005	0,05	20	1	0,000 5
4	0,020	0,20	20	1	0,002 0
10	0,050	0,50	20	1	0,005 0
20	0,100	1,00	20	1	0,010 0

^a Blank test on reagents for calibration curve.

7.4.1.3 Cadmium mass fractions between 0,01 % and 0,10 %

Into each of a series of five 100 ml one-mark volumetric flasks, introduce the volumes of cadmium standard solution (4.5) and copper base solution (4.7) shown in Table 2. Dilute to the mark with water and mix.

Table 2 — Calibration for cadmium mass fractions between 0,01 % and 0,10 %

Cadmium standard solution volume (4.5) ml	Corresponding cadmium mass mg	Corresponding cadmium concentration after final dilution µg/ml	Copper base solution volume (4.7) ml	Corresponding copper mass g	Corresponding cadmium mass fraction of sample %
0 ^a	0	0	10	0,05	0
1	0,005	0,05	10	0,05	0,01
3	0,015	0,15	10	0,05	0,03
6	0,030	0,30	10	0,05	0,06
10	0,050	0,50	10	0,05	0,10

^a Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the cadmium hollow-cathode lamp (5.2) to the atomic absorption spectrometer (5.1), switch on the current and allow it to stabilise. Adjust the wavelength in the region of 228,8 nm to minimum absorbance. Following manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilise. 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.

7.4.3 Spectrometric measurement of the calibration solutions

Aspirate the relevant series of calibration solutions (7.4.1.2 or 7.4.1.3 depending on the expected cadmium content) 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.