SLOVENSKI STANDARD
oSIST prEN 15024-2:2016

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Baker in bakrove zlitine - Določevanje cinka - 2. del: Metoda s plamensko atomsko absorpcijsko spektrometrijo (FAAS)

Copper and copper alloys - Determination of zinc content - Part 2: Flame atomic absorption spectrometric method (FAAS)

Kupfer und Kupferlegierungen - Bestimmung des Zinkgehaltes - Teil 2: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage du zinc - Partie 2 : Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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English Version

Copper and copper alloys - Determination of zinc content - Part 2: Flame atomic absorption spectrometric method (FAAS)

Cuivre et alliages de cuivre - Dosage du zinc - Partie 2 : Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des Zinkgehaltes - Teil 2: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 133.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 15024-2:2016) has been prepared by Technical Committee CEN/TC 133 “Copper and copper alloys”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15024-2:2006.

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 15024-2, *Copper and copper alloys — Determination of zinc content — Part 2: Flame atomic absorption spectrometric method (FAAS)*

This is one of two parts of the standard for the determination of zinc content in copper and copper alloys. The other part is:

prEN 15024-1, *Copper and copper alloys — Determination of zinc content — Part 1: Titrimetric method*

In comparison with EN 15024-2:2006, the following significant changes were made:

a) The upper limit of the zinc mass fraction has been reduced from 6,0 % to 5,0 %;

b) Fluoroboric-nitic acid mixture has been replaced by a solution of hydrochloric and nitric acid for the dissolution of the test portion;

c) The concentrations of the zinc stock solution and the copper base solutions have been modified;

d) The use of a continuum source xenon short arc lamp has been added as an alternative to the use of a zinc hollow-cathode lamp;

e) The precision data have been updated.

1 Scope

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

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

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 and nitric acid solution followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 213,8 nm line emitted by a zinc hollow-cathode lamp or a continuum source xenon short arc lamp.

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 (ρ = 1,19 g/ml)

4.2 Hydrochloric acid solution, 1 + 1

Add 500 ml of hydrochloric acid (4.1) to 500 ml of water.

4.3 Nitric acid, HNO_3 (ρ = 1,40 g/ml)

4.4 Nitric acid solution, 1 + 1

Dilute 500 ml of nitric acid (4.3) in 500 ml of water

4.5 Zinc stock solution, 1 g/l Zn

Weigh (1 ± 0,001) g of zinc (Zn ≥ 99,99 %) and transfer it into a 250 ml beaker. Add 50 ml of hydrochloric acid (4.2). Cover with a watch glass and heat gently to assist dissolution. When dissolution is complete, 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.

1 ml of this solution contains 1 mg of Zn.

4.6 Zinc standard solution, 0,01 g/l Zn

Transfer 5,0 ml of the zinc stock solution (4.5) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,01 mg of Zn.

4.7 Zinc standard solution, 0,001 g/l Zn

Transfer 25,0 ml of the zinc standard solution (4.6) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,001 mg of Zn.

4.8 Copper base solution, 50 g/l Cu

Transfer 50,0 g of zinc-free copper (Cu ≥ 99,99 %) into a 2 000 ml beaker. Add 500 ml of hydrochloric acid (4.1) and, by small fractions, 250 ml of nitric acid (4.3). 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. 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.

4.9 Copper base solution, 2 g/l Cu

Transfer 10,0 ml of copper base solution (4.8) into a 250 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 Zinc hollow-cathode lamp or a continuum source xenon short arc 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 (1 ± 0,001) g of the test sample.

7.1.2 Test portion solution

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 (4.4). 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.

7.1.3 Zinc mass fractions between 0,000 5 % and 0,01 %

Transfer the test portion solution (7.1.2) quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

NOTE The validation inter-laboratory comparison of this method showed that for zinc mass fractions near 0,01 % results are better when the determination is carried out following 7.1.3.

7.1.4 Zinc mass fractions between 0,01 % and 0,10 %

Transfer the test portion solution (7.1.2) quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Transfer 10,0 ml of this solution, into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

NOTE The validation inter-laboratory comparison of this method showed that for zinc mass fractions near 0,1 % results are better when the determination is carried out following 7.1.4.

7.1.5 Zinc mass fractions between 0,10 % and 1,00 %

Transfer the test portion solution (7.1.2) quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Transfer 2,0 ml of this solution, into a 200 ml one-mark volumetric flask and add 10,0 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.

NOTE The validation inter-laboratory comparison of this method showed that for zinc mass fractions near 1,0 % results are better when the determination is carried out following 7.1.5.

7.1.6 Zinc mass fractions between 1,0 % and 5,0 %

Transfer the test portion solution (7.1.2) quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Transfer 2,50 ml of this solution into a 200 ml one-mark volumetric flask and add 10,0 ml of hydrochloric acid (4.1). 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 reference material or a synthetic sample containing a known amount of nickel 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 zinc 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 Zinc mass fractions between 0,000 5 % and 0,01 %

Into each of a series of seven 100 ml one-mark volumetric flasks, introduce the volumes of zinc standard solutions (4.6 or 4.7) and copper base solution (4.8) as shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for zinc mass fractions between 0,000 5 % and 0,01 %

Zinc standard solutions volume (4.6) or (4.7) Ml	Corresponding zinc mass mg	Corresponding zinc concentration after final dilution mg/ml	Copper base solution volume (4.8) ml	Corresponding copper mass mg	Corresponding zinc mass fraction of sample %
0[a]	0	0	20	1 000	0
5 (4.7)	0,005	0,000 05	20	1 000	0,000 5
10 (4.7)	0,010	0,000 1	20	1 000	0,001
20 (4.7)	0,020	0,000 2	20	1 000	0,002
5 (4.6)	0,050	0,000 5	20	1 000	0,005
7 (4.6)	0,070	0,000 7	20	1 000	0,007
10 (4.6)	0,100	0,001	20	1 000	0,01

[a] Blank test on reagents for calibration curve.

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

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of zinc standard solutions (4.6 or 4.7) and copper base solution (4.8) as shown in Table 2. Dilute to the mark with water and mix well.

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

Zinc standard solutions volume (4.6) or (4.7) ml	Corresponding zinc mass mg	Corresponding zinc concentration after final dilution mg/ml	Copper base solution volume (4.8) Ml	Corresponding copper mass mg	Corresponding zinc mass fraction of sample %
0[a]	0	0	2	100	0
10 (4.7)	0,010	0,000 1	2	100	0,01
20 (4.7)	0,020	0,000 2	2	100	0,02
5 (4.6)	0,050	0,000 5	2	100	0,05
7 (4.6)	0,070	0,000 7	2	100	0,07
10 (4.6)	0,100	0,001	2	100	0,10

[a] Blank test on reagents for calibration curve

7.4.1.4 Zinc mass fractions between 0,10 % and 1,00 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of zinc standard solutions (4.6 or 4.7) and copper base solution (4.9) as shown in Table 3. Add 5 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.

Table 3 — Calibration for zinc mass fractions between 0,10 % and 1,00 %

Zinc standard solutions volume (4.6) or (4.7) Ml	Corresponding zinc mass mg	Corresponding zinc concentration after final dilution mg/ml	Copper base solution volume (4.9) Ml	Corresponding copper mass mg	Corresponding zinc mass fraction of sample %
0[a]	0	0	5	10	0
10 (4.7)	0,010	0,000 1	5	10	0,10
20 (4.7)	0,020	0,000 2	5	10	0,20
5 (4.6)	0,050	0,000 5	5	10	0,50
7 (4.6)	0,070	0,000 7	5	10	0,70
10 (4.6)	0,100	0,001	5	10	1,00

[a] Blank test on reagents for calibration curve

7.4.1.5 Zinc mass fractions between 1,0 % and 5,0 %

Into each of a series of six 200 ml one-mark volumetric flasks, introduce the volumes of zinc standard solution (4.6) and copper base solution (4.9) as shown in Table 4. Add 10 ml of hydrochloric acid (4.1). Dilute to the mark with water and mix well.