SLOVENSKI STANDARD

SIST EN 14942-2:2006

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Baker in bakrove zlitine - Ugotavljanje vsebnosti arzena - 2. del: Metoda FAAS

Copper and copper alloys - Determination of arsenic content - Part 2: FAAS method

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

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

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English Version

Copper and copper alloys - Determination of arsenic content - Part 2: FAAS method

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

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

This European Standard was approved by CEN on 15 May 2006.

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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.

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Foreword

This document (EN 14942-2:2006) 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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.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 14942-2, *Copper and copper alloys — Determination of arsenic content — Part 2: FAAS method*

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

prEN 14942-1, *Copper and copper alloys — Determination of arsenic content — Part 1: Spectrometric method*

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.

1 Scope

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

The method is applicable to products having arsenic mass fractions between 0,01 % and 1,5 %.

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*

NOTE Informative references to documents used in the preparation of this standard, and cited at the appropriate places in the text, are listed in the Bibliography.

3 Principle

Dissolution of a test portion in hydrochloric acid and hydrogen peroxide followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 193,7 nm line emitted by an arsenic hollow-cathode or electrodeless discharge lamp.

4 Reagents and materials

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 (ρ = 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, H_2O_2 30 % (mass fraction) solution.

4.5 Potassium hydroxide, KOH 20 % solution

Weigh 20,0 g of potassium hydroxide pellets and dissolve gently in 50 ml of water. Cool to room temperature and dilute to 100 ml with water.

4.6 Arsenic stock solution, 1,000 g/l As

Weigh (1,320 ± 0,001) g of primary standard arsenious oxide (As_2O_3 purity ≥ 99,5 %) and dissolve it in 25 ml of the potassium hydroxide solution (4.5). Transfer the solution quantitatively into a 1 000 ml one-mark polyethylene volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of As.

4.7 Arsenic standard solution, 0,050 g/l As

Transfer 10,0 ml of the arsenic stock solution (4.6) into a 200 ml one-mark polyethylene volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,050 mg of As.

4.8 Arsenic standard solution, 0,500 g/l As

Transfer 50,0 ml of the arsenic stock solution (4.6) into a 100 ml one-mark polyethylene volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,500 mg of As.

4.9 Copper base solution, 50 g/l Cu

Weigh 31,294 g of copper II oxide (CuO ≥ 98 %, As ≤ 0,000 2 %) or 25 g copper (Cu ≥ 99,90 %, As ≤ 0,000 2 %) and transfer into a 800 ml beaker. Dissolve it gently with 100 ml of hydrochloric acid solution (4.3) previously cooled. Add slowly hydrogen peroxide (4.4) in small portions while cooling (approximately 50 ml total) until the dissolution is complete. Cover and heat to the boiling point. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water. Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, add 250 ml of hydrochloric acid solution (4.3), dilute to the mark with water and mix well.

5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Atomic absorption spectrometer, fitted with an air/acetylene burner.

5.3 Arsenic hollow-cathode or electrodeless discharge 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 Arsenic mass fractions less than 0,10 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 15 ml of hydrochloric acid solution (4.3) and, drop by drop, 10 ml of hydrogen peroxide (4.4). Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat the solution to boil gently for 1 min. Cool to room temperature, transfer the solution quantitatively into a 100 ml volumetric flask, dilute to the mark with water and mix well.

7.1.3 Arsenic mass fractions between 0,10 % and 1,5 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 15 ml of hydrochloric acid solution (4.3) and, drop by drop, 10 ml of hydrogen peroxide (4.4). Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat the solution to boil gently for 1 min. Cool to room temperature, transfer quantitatively the solution into a 200 ml volumetric flask, add 10 ml of the hydrochloric solution (4.3). 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 omitting 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 arsenic 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

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 standard 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 arsenic concentration of the calibration solutions shall be adjusted to suit the sensitivity of the apparatus used, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Arsenic 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 the arsenic standard solution (4.7) and of the copper base solution (4.9) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for arsenic mass fractions between 0,01 % and 0,10 %

Arsenic standard solution volume (4.7) ml	Corresponding arsenic mass mg	Corresponding arsenic concentration after final dilution mg/ml	Copper base solution volume (4.9) ml	Corresponding copper mass g	Corresponding arsenic mass fraction of sample %
0[a]	0	0	20	1,000	0
2	0,10	0,001 0	20	1,000	0,010
5	0,25	0,002 5	20	1,000	0,025
10	0,50	0,005 0	20	1,000	0,050
15	0,75	0,007 5	20	1,000	0,075
20	1,00	0,010	20	1,000	0,100

[a] Blank test on reagents for calibration curve.

7.4.1.3 Arsenic mass fractions between 0,10 % and 1,5 %

Into each of a series of eight 100 ml one-mark volumetric flasks, introduce the volumes of the arsenic standard solution (4.8) and of the copper base solution (4.9) shown in Table 2. Dilute to the mark with water and mix well.

Table 2 — Calibration for arsenic mass fractions between 0,10 % and 1,5 %

Arsenic standard solution volume (4.8) ml	Corresponding arsenic mass mg	Corresponding arsenic concentration after final dilution mg/ml	Copper base solution volume (4.9) ml	Corresponding copper mass g	Corresponding arsenic mass fraction of sample %
0[a]	0	0	10	0,500	0
1	0,5	0,005 0	10	0,500	0,10
2	1	0,010	10	0,500	0,20
4	2	0,020	10	0,500	0,40
6	3	0,030	10	0,500	0,60
8	4	0,040	10	0,500	0,80
10	5	0,050	10	0,500	1,00
15	7,5	0,075	10	0,500	1,50

[a] Blank test on reagents for calibration curve.