SLOVENSKI STANDARD
SIST-TS CEN/TS 14938-2:2006

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Baker in bakrove zlitine - Določ evanje bizmuta - 2. del: Metoda FAAS

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

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

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

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English Version

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

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

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

This Technical Specification (CEN/TS) was approved by CEN on 23 July 2006 for provisional application.

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Foreword

This document (CEN/TS 14938-2:2006) 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 14938-2, *Copper and copper alloys — Determination of bismuth content — Part 2: FAAS method*

This is one of two parts of the standard/technical specification for the determination of bismuth content in copper and copper alloys. The other part is under preparation:

…, *Copper and copper alloys — Determination of bismuth content — Part 1: Spectrophotometric method*

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.

1 Scope

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

The method is applicable to products having bismuth mass fractions between 0,01 % and 0,25 %.

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*

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 a mixture of hydrochloric acid/hydrogen peroxide and nitric acid solutions followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 223,1 nm line emitted by a bismuth 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 Nitric acid, HNO_3 (ρ = 1,40 g/ml).

4.4 Hydrogen peroxide, H_2O_2 30 % (mass fraction) solution.

4.5 Bismuth stock solution, 1,000 g/l Bi

Weigh (0,25 ± 0,001) g of bismuth (Bi ≥ 99,9999 %) and transfer it into a 250 ml conical flask. Add 50 ml of nitric acid (4.3). Heat gently until the bismuth is dissolved and then bring to the boiling point until the nitrous fumes have been expelled. Cool to room temperature, transfer the solution quantitatively into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

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

4.6 Bismuth standard solution, 0,100 g/l Bi

Transfer 25,0 ml of the bismuth stock solution (4.5) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of Bi.

4.7 Copper base solution, 20 g/l Cu

Into a 600 ml tall beaker weigh 10,0 g of bismuth-free copper (Bi ≤ 0,005 %). Add 50 ml of distilled water and, in small portions, 50 ml of nitric acid (4.3). Cover and heat gently until the copper is completely dissolved, then continue heating 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, dilute to the mark with water and mix well.

1 ml of this solution contains 0,02 g of Cu.

5 Apparatus

5.1 Ordinary laboratory apparatus.

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

5.3 Bismuth 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 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 10 ml of distilled water and 0,5 ml of nitric acid (4.3). Add 0,5 ml of hydrogen peroxide (4.4) and 20 ml of hydrochloric acid (4.2). Cover with a watch glass and heat gently until the test portion is completely dissolved, then continue heating to the boiling point. Cool to room temperature. Wash the beaker cover and the sides of the beaker with water.

Transfer the dissolved test portion into a 100 ml one-mark volumetric flask. 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 standard material or a synthetic sample containing a known amount of bismuth 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, 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 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 bismuth 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 Bismuth mass fractions between 0,01 % and 0,05 %

Into each of a series of five 100 ml one-mark volumetric flasks, introduce 20,0 ml of hydrochloric acid (4.2) and the volumes of the bismuth standard solution (4.6) and of the copper base solution (4.7) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for bismuth mass fractions between 0,01 % and 0,05 %

Bismuth standard solution volume (4.6) ml	Corresponding bismuth mass mg	Corresponding bismuth concentration after final dilution mg/ml	Copper base solution volume (4.7) ml	Corresponding copper mass g	Corresponding bismuth mass fraction of sample %
0[a]	0	0	50	1,000	0
1	0,10	0,001 0	50	1,000	0,010
2	0,20	0,002 0	50	1,000	0,020
3	0,30	0,003 0	50	1,000	0,030
5	0,50	0,005 0	50	1,000	0,050

[a] Blank test on reagents for calibration curve.

7.4.1.3 Bismuth mass fractions between 0,050 % and 0,25 %

Into each of a series of five 100 ml one-mark volumetric flasks, introduce 20,0 ml of hydrochloric acid (4.2) and the volumes of the bismuth stock solution (4.5) and of the copper base solution (4.7) shown in Table 2. Dilute to the mark with water and mix well.

Table 2 — Calibration for bismuth mass fractions between 0,05 % and 0,25 %

Bismuth stock solution volume (4.5) ml	Corresponding bismuth mass mg	Corresponding bismuth concentration after final dilution mg/ml	Copper base solution volume (4.7) ml	Corresponding copper mass g	Corresponding bismuth mass fraction of sample %
0[a]	0	0	50	1,00	0
0,5	0,50	0,005	50	1,00	0,050
1,0	1,00	0,010	50	1,00	0,10
1,5	1,50	0,015	50	1,00	0,15
2,5	2,50	0,025	50	1,00	0,25

[a] Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the bismuth hollow-cathode or electrodeless discharge lamp (5.3) into the atomic absorption spectrometer (5.2), switch on the current and allow to stabilize. 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.

7.4.3 Spectrometric measurement of the calibration solutions

Aspirate the relevant series of calibration solutions (see 7.4.1.2 or 7.4.1.3 depending on the expected bismuth 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.

7.4.4 Calibration curve

Establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or 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

7.5.3.1 Use of the calibration curve

Repeat the measurements and derive the concentration directly using the appropriate calibration curve.