SLOVENSKI STANDARD
SIST-TS CEN/TS 15616:2009

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

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

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

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

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77.120.30	Baker in bakrove zlitine	Copper and copper alloys

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English Version

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

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

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

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Foreword

This document (CEN/TS 15616:2009) has been prepared by Technical Committee CEN/TC 133 “Copper and copper alloys”, the secretariat of which is held by DIN.

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1 Scope

This document specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the cadmium content of copper and copper alloys in the form of castings or unwrought or wrought products.

The method is applicable to products having cadmium mass fractions between 0,002 % and 2,0 %.

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 a fluoroboric-nitric acid mixture followed, after suitable dilution, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Determination of the cadmium content by measuring the absorption of the 228,8 nm line emitted by a cadmium hollow-cathode lamp.

4 Reagents and materials

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

4.1 Boric acid, H_3BO_3 solution 40 g/l.

4.2 Hydrofluoric acid, HF 40 % (ρ = 1,13 g/ml).

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

4.4 Fluoroboric-nitric acid mixture

Mix together 300 ml of boric acid solution (4.1), 30 ml of hydrofluoric acid (4.2), 500 ml of nitric acid (4.3) and 150 ml of water.

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

Transfer (1 ± 0,001) g of cadmium (Cd ≥ 99,99 %) into a 250 ml tall-form beaker. Add 10 ml of water and 5 ml of nitric acid (4.3), cover with a watch glass and heat gently (if necessary) until the cadmium is completely dissolved. Boil gently to expel nitrous fumes. Allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Add 100 ml of water and 20 ml of nitric acid (4.3). Dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cd.

4.6 Cadmium standard solution, 0,050 g/l Cd

Transfer 10,0 ml of the cadmium stock solution (4.5) into a 200 ml one-mark volumetric flask. Add 100 ml of water and 20 ml of nitric acid (4.3). Dilute to the mark with water and mix well. Prepare this solution immediately prior to use.

1 ml of this solution contains 50,0 µg of Cd.

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

Transfer 20,0 ml of the cadmium standard solution (4.6) into a 200 ml one-mark volumetric flask. Add 100 ml of water and 18 ml of nitric acid (4.3). Dilute to the mark with water and mix well. Prepare this solution immediately prior to use.

1 ml of this solution contains 5,0 µg of Cd.

4.8 Copper base solution, 20 g/l Cu

Weigh 20,0 g of pure copper containing not more than 0,000 5 % Cd into a 1 000 ml beaker (5.1). Add 800 ml of fluoroboric-nitric acid mixture (4.4). Heat gently until the copper is completely dissolved, then boil until nitrous fumes have been expelled. In the case of polyethylene or polypropylene beakers, use a water bath for heating. 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 well.

50 ml of this solution contain 1 g of Cu and 40 ml of solution (4.4).

5 Apparatus

5.1 Polytetrafluorethylene, polypropylene or low-pressure polyethylene beakers, capacity 250 ml and 1 000 ml.

5.2 Flasks, polytetrafluorethylene , polyethylene or polypropylene, capacities 250 ml and 600 ml.

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

5.4 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

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 PTFE, polypropylene or low-pressure polyethylene beaker (5.1). Add 40 ml of the attack reagent (4.4). Cover with a watch glass and heat gently until the test portion is dissolved, then heat at a temperature of approximately 90 °C until nitrous fumes have been expelled. Wash the cover and the sides of the beaker with water, and cool.

7.1.3 Cadmium mass fractions between 0,002 % and 0,075 %

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.

7.1.4 Cadmium mass fractions between 0,05 % and 0,75 %

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.

7.1.5 Cadmium mass fractions between 0,5 % and 2,0 %

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 20,0 ml of this solution into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 10 ml of this solution 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 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 a standard material or a synthetic sample containing a known amount of cadmium and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Establishment of the calibration solutions

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

In all cases, copper, fluoride, borate and nitrate concentrations, and the pH-values of 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 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 (4.8) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Overcompensation 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 0,002 % and 0,075 %

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

7.4.1.3 Cadmium mass fractions between 0,05 % and 0,75 %

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

7.4.1.4 Cadmium mass fractions between 0,5 % and 2,0 %

Into each of a series of five 100 ml one-mark volumetric flasks, introduce the volumes of cadmium standard solution (4.6) and of copper base solution (4.8) as shown in Table 3. Dilute to the mark with water and mix well.