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Koppar och kopparlegeringar – Bestämning av järnhalt – Del 2: Flamatomär absorptionsspektrometrimetod (FAAS)

Copper and copper alloys – Determination of iron content – Part 2: Flame atomic absorption spectrometry method (FAAS)

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EUROPEAN STANDARD

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EUROPÄISCHE NORM

EN 15690-2

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ICS 77.040.30; 77.120.30

English Version

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

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

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

This European Standard was approved by CEN on 26 December 2008.

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Foreword

This document (EN 15690-2:2009) 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of an identical text or by endorsement, at the latest by August 2009, and conflicting national standards shall be withdrawn at the latest by August 2009.

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.

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 15690-2, *Copper and copper alloys — Determination of iron content — Part 2: Flame atomic absorption spectrometric method (FAAS)*

This is one of two Parts of the standard for the determination of iron content in copper and copper alloys. The other Part is:

EN 15690-1, *Copper and copper alloys — Determination of iron content — Part 1: Titrimetric method*

Part 1 will be the subject of future work.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, 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 Part of this European Standard specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the iron content of copper and copper alloys in the form of castings or unwrought or wrought products.

The method is applicable to products having iron mass fractions between 0,005 % and 5,0 %.

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 hydrochloric and nitric acid mixture followed, after suitable dilution and the addition of lanthanum chloride to mask the effect of interfering ions, by aspiration of the test solution into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 248,3 nm or the 372,0 nm line emitted by an iron hollow-cathode 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.

Avoid any contamination with iron during the mechanical preparation steps.

4.2 **Hydrochloric acid,** HCl (ρ = 1,19 g/ml)

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

4.4 **Nitric acid,** (1 + 1)

Add 500 ml of nitric acid (4.3) into 500 ml of water.

4.5 **Hydrofluoric acid,** HF (ρ = 1,13 g/ml)

WARNING — Hydrofluoric acid is a hazardous substance. Care shall be taken and it shall be used under an efficient fume hood.

4.6 Lanthanum(III) chloride solution, 100 g/l

Weigh 100 g of lanthanum(III) chloride ($LaCl_3 \cdot 7H_2O$) in a 600 ml beaker, transfer it into a 1 000 ml one-mark volumetric flask and dissolve it with water. Dilute to the mark with water and mix well.

4.7 Iron stock solution, 0,5 g/l Fe

a) Weigh (0,5 ± 0,001) g of high purity iron and transfer it into a 250 ml beaker. Dissolve it in 50 ml of hydrochloric acid (4.2), 25 ml water and 2,5 ml nitric acid (4.3). 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 well; or

b) Weigh (0,715 ± 0,000 1) g of high purity iron(III) oxide (Fe_2O_3), previously dried and transfer it into a 250 ml beaker. Add 50 ml of hydrochloric 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 well.

1 ml of this solution contains 0,5 mg of Fe.

4.8 Iron standard solution, 0,05 g/l Fe

Transfer 20,0 ml of iron stock solution (4.7) into a 200 ml one-mark volumetric flask. Add 5 ml of hydrochloric acid (4.2), dilute to the mark with water and mix well.

1 ml of this solution contains 0,05 mg of Fe.

4.9 Iron standard solution, 0,01 g/l Fe

Transfer 10,0 ml of iron stock solution (4.7) into a 500 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.2), dilute to the mark with water and mix well.

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

4.10 Copper base solution, 20 g/l Cu

Transfer (10 ± 0,01) g of iron-free copper (Cu ≥ 99,95 %) after etching into a 600 ml beaker. Add 100 ml of hydrochloric acid (4.2) and, cautiously, 100 ml of nitric acid solution (4.4). 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 well.

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

4.11 Copper base solution, 2,0 g/l Cu

Transfer quantitatively 25 ml of copper base solution (4.10) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 2,0 mg of Cu.

5 Apparatus

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

5.2 Iron 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 beaker. Add 10 ml of hydrochloric acid (4.2) 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. Allow to cool. If undissolved matter remains, indicating the presence of silicon, filter the solution. Place the filter paper and contained salts in a platinum crucible and ash, taking care that the filter does not flame. Calcine at about 550 °C. Cool and add 5 ml of hydrofluoric acid (4.5) and five drops of nitric acid (4.3). Evaporate to dryness and calcine again for several minutes at about 700 °C to completely volatilize the silicon. Cool, and then dissolve the residue with the least possible volume of nitric acid solution (4.4). Filter, if necessary, and add this filtrate quantitatively to the original filtrate.

7.1.3 Iron mass fractions between 0,005 % and 0,025 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Add 10 ml of the lanthanum(III) chloride solution (4.6) and 1 ml of hydrochloric acid (4.2), dilute to the mark with water and mix well.

7.1.4 Iron mass fractions between 0,025 % and 0,5 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 20 ml of this solution into a 100 ml one-mark volumetric flask. Add 10 ml of the lanthanum(III) chloride solution (4.6) and 1 ml of hydrochloric acid (4.2), dilute to the mark with water and mix well.

7.1.5 Iron mass fractions between 0,5 % and 5 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix well. Transfer 5 ml of this solution into a 250 ml one-mark volumetric flask. Add 25 ml of the lanthanum(III) chloride solution (4.6) and 1 ml of hydrochloric acid (4.2), 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, by pure copper for the test portion (7.1.1). Correct the result obtained from the determination in accordance with the result of the blank.

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 iron 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, salts concentration 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 (4.10 and 4.11) 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 iron 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.