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

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

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

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

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Cuivre et alliages de cuivre - Détermination de manganèse
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Kupfer und Kupferlegierungen - Bestimmung des
Mangangehaltes - Teil 2:
Flammenatomabsorptionsspektrometrisches Verfahren
(FAAS)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 15703-2:2014) 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of an identical text or by endorsement, at the latest by June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.

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 15703-2, *Copper and copper alloys — Determination of manganese content — Part 2: Flame atomic absorption spectrometric method (FAAS)*

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

CEN/TS 15703-1, *Copper and copper alloys — Determination of manganese content — Part 1: Spectrophotometric 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, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

EN 15703-2:2014 (E)**1 Scope**

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

The method is applicable to products having manganese mass fractions between 0,001 0 % and 6,0 %.

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 acid and nitric acid followed, after suitable dilution and the addition of lanthanum chloride to mask the effect of interfering ions, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 279,5 nm or 403,1 nm line emitted by a manganese hollow-cathode 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 ($\rho = 1,19$ g/ml)**4.2 Nitric acid, HNO₃ ($\rho = 1,40$ g/ml)****4.3 Hydrofluoric acid, HF ($\rho = 1,13$ g/ml)**

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

4.4 Nitric acid solution, 1 + 1

Add 500 ml of nitric acid (4.2) to 500 ml of water.

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

Dissolve 100 g of lanthanum(III) chloride ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) in a 600 ml beaker with water and transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

4.6 Sulphuric acid, H₂SO₄ ($\rho = 1,84$ g/ml)

4.7 Sulphuric acid solution, 1 + 9

Add 20 ml of sulphuric acid (4.6) in 180 ml water.

4.8 Manganese stock solution, 1,0 g/l Mn

4.8.1 Cleaning of manganese metal

Transfer several grams of electrolytic manganese (purity > 99,9 %) into a 250 ml beaker containing about 150 ml of sulphuric acid solution (4.7). Stir, then allow the manganese to settle for several minutes. Decant and discard the sulphuric acid solution. Rinse several times with water and finally with acetone. Dry the metal for about 2 min at 100°C and cool in a desiccator.

4.8.2 Preparation of stock solution

Weigh, $(1 \pm 0,001)$ g, of manganese cleaned as in 4.8.1 and transfer it into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.1) and 20 ml of the nitric acid solution (4.4). 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 1,0 mg of Mn.

4.9 Manganese standard solution, 0,10 g/l

Transfer 20,0 ml of manganese stock solution (4.8) into a 200 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 0,10 mg of Mn.

4.10 Manganese standard solution, 0,010 g/l

Transfer 5,0 ml of manganese stock solution (4.8) to a 500 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 0,010 mg of Mn.

4.11 Copper matrix solution, 20 g/l Cu

Transfer $(10 \pm 0,01)$ g of manganese-free copper ($\text{Cu} \geq 99,95\%$) into a 600 ml beaker. Add 100 ml of hydrochloric acid (4.1) and, cautiously, 100 ml of the 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.

4.12 Copper matrix solution, 2 g/l Cu

Transfer 50,0 ml of the copper matrix solution (4.11) into a 500 ml one-mark volumetric flask. Add 90 ml of hydrochloric acid (4.1) and 90 ml of the nitric acid solution (4.4). Dilute to the mark with water and mix well.

5 Apparatus

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

EN 15703-2:2014 (E)**5.2 Manganese 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 ($1 \pm 0,001$) 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 (4.1) 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. Allow to cool. If undissolved matter remains, indicating the presence of silicon, filter the solution. Place the filter and retained residues in a platinum crucible and ash, taking care that the filter does not flame. Calcine at about 550 °C. Allow to cool and add 5 ml of hydrofluoric acid (4.3) and five drops of nitric acid (4.2). 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 Manganese mass fractions between 0,001 % and 0,020 %

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 (4.5), dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions between 0,015 % and 0,020 % results are better when the determination is carried out following 7.1.3.

7.1.4 Manganese mass fractions between 0,020 % and 0,50 %

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 5,0 ml of this solution into a 100 ml one-mark volumetric flask. Add 4,5 ml of hydrochloric acid (4.1), 4,5 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

7.1.5 Manganese mass fractions between 0,50 % and 2,0 %

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 10,0 ml of this solution into a 100 ml one-mark volumetric flask. Add 9 ml of hydrochloric acid (4.1), 9 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions near 0,5 % results are better when the determination is carried out following 7.1.5.

7.1.6 Manganese mass fractions between 2,0 % and 6,0 %

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 2,5 ml of this solution into a 100 ml one-mark

volumetric flask. Add 2,3 ml of hydrochloric acid (4.1), 2,3 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions near 2,0 % results are better when the determination is carried out following 7.1.6.

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 a reference material or a synthetic sample containing a known amount of manganese 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 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 matrix solution added (4.11 or 4.12) 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 matrix solution shall be decreased to match the copper content of the test sample in solution.

The manganese 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 Manganese mass fractions between 0,001 0 % and 0,020 %

Into each of a series of seven 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.10) and copper matrix solution (4.11) shown in Table 1. Introduce also 10 ml of lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.