SLOVENSKI STANDARD
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Baker in bakrove zlitine - Določevanje mangana - 1. del: Spektrofotometrična metoda

Copper and copper alloys - Determination of manganese content - Part 1: Spectrophotometric method

Kupfer und Kupferlegierungen - Bestimmung des Mangangehaltes - Teil 1: Spektrophotometrisches Verfahren

Cuivre et alliages de cuivre - Dosage du manganese - Partie 1 : Méthode spectrophotométrique

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Copper and copper alloys - Determination of manganese content - Part 1: Spectrophotometric method

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Contents

Page

Foreword

This document (CEN/TS 15703-1:2009) 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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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:

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: 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 Technical Specification specifies a spectrophotometric method for the determination of the manganese content of copper and copper alloys in the form of castings or unwrought or wrought products.

The method is applicable to products having manganese mass fractions between 0,025 % and 6,25 %.

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*

3 Principle

Dissolution of a test portion in fluoroboric-nitric acid mixture. Oxidation of manganese to permanganic acid by potassium periodate. Spectrophotometric determination in comparison with a background colour prepared by selective reduction of permanganic acid by sodium nitrite.

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 (40 g/l solution)

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

4.3 Nitric acid solution (1 + 3)

Add 25 ml of nitric acid (4.2) to 75 ml of water.

4.4 Hydrofluoric acid, HF 40 % (ρ = 1,14 g/ml)

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

4.5 Sulphuric acid, H_2SO_4 (ρ = 1,84 g/ml)

4.6 Sulphuric acid solution (1 + 3)

Add 25 ml of sulphuric acid (4.5) to 75 ml of water.

4.7 Potassium periodate, KIO_4 crystals

4.8 Potassium periodate solution, KIO_4 50 g/l

Dissolve 5 g of potassium periodate (4.7) in 100 ml of nitric acid solution (4.3).

4.9 Sodium nitrite, $NaNO_2$ solution, 20 g/l

Dissolve 2,0 g of sodium nitrite in water. Transfer this solution into a 100 ml volumetric flask. Make up to 100 ml and mix. The solution shall be freshly prepared.

4.10 Fluoroboric-nitric acid mixture

Using a polypropylene stirrer in a 1 000 ml polypropylene tall form beaker, mix well:

a) 150 ml of water;

b) 300 ml of boric acid solution (4.1);

c) 50 ml of nitric acid (4.2);

d) 30 ml of hydrofluoric acid (4.4).

4.11 Dilution solution

To 1 000 ml of boric acid solution (4.1) add 5,5 ml of sulphuric acid (4.5). Bring to the boil and add a few crystals of potassium periodate (4.7). Cool and transfer the solution into a stoppered 1 000 ml conical flask.

4.12 Manganese stock solution, 1 g/l Mn

Place a few grams of electrolytic manganese (Mn ≥ 99,9 %) in a 250 ml glass beaker containing 60 ml to 80 ml of sulphuric acid solution (4.6) and 100 ml of water. Stir, and after a few minutes, decant the acid solution and wash the metal with water several times, decanting the liquid each time. Place the metal in acetone and stir. Decant the acetone and dry the metal and allow it to cool in a desiccator. In a 600 ml tall form beaker, dissolve (1 ± 0,001) g of the cleaned manganese in 40 ml of sulphuric acid solution (4.6) and about 80 ml of water. When dissolution is complete, boil the solution for several minutes. Cool and transfer the solution to a 1 000 ml volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mn.

4.13 Manganese standard solution, 0,1 g/l Mn

Transfer 20 ml of the manganese stock solution (4.12) to a 200 ml volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,1 mg of Mn.

5 Apparatus

5.1 Ordinary laboratory apparatus

5.2 Spectrophotometer, fitted with cells of optical path lengths of 1 cm, 2 cm and 4 cm

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 (0,4 ± 0,001) g of the test sample into a 300 ml conical flask.

7.1.2 Test portion solution

7.1.2.1 General

Dissolve the test portion (7.1.1) with 50 ml of the fluoroboric-nitric acid mixture (4.10). Warm, if necessary, to accelerate the attack. When dissolution is complete, add 20 ml of water. Boil for 5 min to remove the oxides of nitrogen.

7.1.2.2 Mass fractions less than 0,5 %

Introduce 5 ml of potassium periodate solution (4.8) into the boiling solution (7.1.2.1). Maintain at boiling for 5 min, then immerse the conical flasks in a boiling water bath for 20 min. Cool and transfer the solution to a 100 ml volumetric flask. Use the dilution solution (4.11) for rinsing and making up to the mark and mix well.

7.1.2.3 Mass fractions between 0,5 % and 2,5 %

Cool the solution (7.1.2.1) and transfer to a 100 ml volumetric flask. Dilute to the mark with water and mix well. Transfer a 20 ml aliquot to a 300 ml conical flask, then add 40 ml of fluoroboric-nitric acid mixture (4.10) and 10 ml of water in order to obtain the same condition of dilution as in the preceding case.

Boil for 5 min and introduce 5 ml of potassium periodate solution (4.8) into the boiling solution. Maintain at boiling for 5 min, then immerse the conical flasks in a boiling water bath for 20 min. Cool and transfer the solution to a 100 ml volumetric flask. Use the dilution solution (4.11) for rinsing and making up to the mark and mix well.

7.1.2.4 Mass fractions between 2 % and 6,25 %

Cool the solution (7.1.2.1) and transfer to a 250 ml volumetric flask. Dilute to the mark with water and mix well. Transfer a 20 ml aliquot to a 300 ml conical flask, then add 46 ml of fluoroboric-nitric acid mixture (4.10) and 10 ml of water in order to obtain the same condition of dilution as in the preceding case.

Boil for 5 min and introduce 5 ml of potassium periodate solution (4.8) into the boiling solution. Maintain at boiling for 5 min, then immerse the flasks in a boiling water bath for 20 min. Cool and transfer the solution to a 100 ml volumetric flask. Use the dilution solution (4.11) for rinsing and making up to the mark 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. 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 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

Into each of a series of seven 300 ml conical flasks, introduce approximately the volume of water and exactly the volume of manganese standard solution (4.13) as shown in Table 1.

Add 50 ml of the fluoroboric-nitric acid mixture (4.10). Boil for 5 min in order to eliminate any oxides of nitrogen which may be present.

Table 1 — Calibration for manganese mass fraction between 0,025 % and 6,25 %

Water volume	Manganese standard solution (4.13)		Corresponding manganese mass fraction of test sample %		
	Volume	Mass of Mn contained	For a test portion of		
ml	ml	mg	0,400 g	0,080 g	0,032 g
20	0[a]	0	0	0	0
19	1	0,1	0,025	0,125	0,312 5
18	2	0,2	0,050	0,250	0,625
16	4	0,4	0,1	0,5	1,25
14	6	0,6	0,15	0,75	1,875
10	10	1	0,25	1,25	3,125
0	20	2	0,50	2,50	6,25

[a] Blank test on reagents for calibration curve.

Introduce 5 ml of potassium periodate solution (4.8) into each of the boiling solutions. Maintain at boiling for 5 min, then immerse the conical flasks in a boiling water bath for 20 min. Cool and transfer to a 100 ml volumetric flask. Use the dilution solution (4.11) for rinsing and making up to the mark and mix well.