SLOVENSKI STANDARD
SIST EN 16117-2:2014

01-julij-2014

Baker in bakrove zlitine - Določevanje bakra - 2. del: Elektrolitsko ugotavljanje bakra v materialih z deležem bakra, večjim od 99,80 %

Copper and copper alloys - Determination of copper content - Part 2: Electrolytic determination of copper in materials with copper content higher than 99,80 %

Kupfer und Kupferlegierungen - Bestimmung des Kupfergehaltes - Teil 2: Elektrolytische Bestimmung von Kupfer in Werkstoffen mit einem Kupfergehalt größer als 99,80 %

Cuivre et alliages de cuivre - Détermination de la teneur en cuivre - Partie 2: Détermination par électrogravimétrie de la teneur en cuivre dans les matériaux ayant une teneur en cuivre supérieure à 99,80 %

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ICS:

77.120.30	Baker in bakrove zlitine	Copper and copper alloys

SIST EN 16117-2:2014 **en,fr,de**

EUROPEAN STANDARD
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Copper and copper alloys - Determination of copper content - Part 2: Electrolytic determination of copper in materials with copper content higher than 99,80 %

Cuivre et alliages de cuivre - Détermination de la teneur en cuivre - Partie 2 : Détermination par électrogravimétrie du cuivre dans les matériaux ayant une teneur en cuivre supérieure à 99,80 %

Kupfer und Kupferlegierungen - Bestimmung des Kupfergehaltes - Teil 2: Elektrolytische Bestimmung von Kupfer in Werkstoffen mit einem Kupfergehalt größer als 99,80 %

This European Standard was approved by CEN on 20 October 2012.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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 United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Contents

Foreword

This document (EN 16117-2:2012) 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 May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

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 16117-2, *Copper and copper alloys — Determination of copper content — Part 2: Electrolytic determination of copper in materials with copper content higher than 99,80 %.*

This is one of two parts of the standard for the determination of the copper content of copper and copper alloys. The other part is:

EN 16117-1, *Copper and copper alloys — Determination of copper content — Part 1: Electrolytic determination of copper in materials with copper content less than 99,85 %*.

According to the CEN/CENELEC Internal Regulations, the national standards organisations 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.

1 Scope

This European Standard specifies an electrolytic method for the determination of the copper content of unalloyed copper materials with a copper content higher than 99,80 % (mass fraction) in the form of castings, wrought and unwrought products.

Silver, if present, is co-deposited and is reported as copper. Approximately one-half of any selenium and tellurium present will co-deposit. Bismuth, if present, also interferes.

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 a nitric and sulphuric acid mixture. Electrolytic deposition of the copper on a platinum cathode of known weight with subsequent weighing of the cathode together with the electrodeposited copper. Determination of residual copper in the spent electrolyte by atomic absorption spectrometry.

4 Reagents

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

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

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

4.3 Sulphuric acid-nitric acid mixture

While stirring, slowly add 300 ml of sulphuric acid (4.1) to 750 ml of water. Cool to ambient temperature. While stirring, cautiously add 210 ml of nitric acid (4.2).

4.4 Sulphamic acid, $H(NH_2)SO_3$

4.5 Sulphamic acid solution, 100 g/l $H(NH_2)SO_3$

Dissolve 10 g of sulphamic acid (4.4) in water and dilute to 100 ml. Prepare freshly before using.

4.6 Alcohol

Ethanol: 95 % ± 0,2 %, methanol: min. 99,9 %, other alcohols: higher than 99,9 %.

5 Apparatus

5.1 Current source

Preferably use a 6 V accumulator. If a rectifier is to be used, an additional buffer battery is recommended.

5.2 Electrolysis equipment

5.2.1 Platinum cathode, Winkler type[1], made preferably from gauze containing approximately 400 meshes per square centimetre, woven from wire of diameter approximately 0,2 mm. The cathode shall be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. The diameter of the cylinder shall be 30 mm to 50 mm and the height 40 mm to 60 mm. The stem shall be made from a platinum alloy wire such as platinum-iridium, platinum-rhodium or platinum-ruthenium, having a diameter of approximately 1,3 mm, flattened and welded the entire length of the gauze. The overall height of the cathode shall be approximately 130 mm. The cathode shall be sandblasted.

5.2.2 Spiral anode, made of platinum alloy wire of minimum diameter 1 mm, formed into a spiral of seven turns, having a height of approximately 50 mm and a diameter of 12 mm, the overall height being approximately 130 mm. The spiral section shall be sandblasted.

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 Test portion

Weigh (5,0 ± 0,001) g of the test sample.

7.2 Determination

7.2.1 Transfer the test portion (7.1) to a 400 ml tall-form beaker. Add 45 ml of the sulphuric acid-nitric acid mixture (4.3) by small portions and then cover with a watch glass. Cool if required to prevent the reaction from becoming violent. When the reaction has subsided, heat moderately (80 °C to 90 °C) until dissolution is complete. Continue heating at approximately 90 °C, without boiling, to completely expel the oxides of nitrogen (more than 1 h may be necessary). Cool slightly and carefully wash down the watch glass and the sides of the beaker with water. Add 10 ml of the sulphamic acid solution (4.5), stir and dilute to 175 ml to 200 ml.

7.2.2 Weigh the cathode (5.2.1) to the nearest 0,1 mg.

7.2.3 Position the electrodes (5.2.1 and 5.2.2) in the solution and add water so that the gauze is completely immersed. Cover the beaker with two halves of a watch glass, one of which has two indentations through which the electrode stems may pass. Electrolyze at a current of about 1 A to 3 A, slowly stirring the electrolyte by a magnetic stirrer, avoiding turbulence (see Note). When the solution becomes colourless, wash the cover, the electrode stem and the sides of the beaker. Add 10 ml of sulphamic acid solution (4.5) and continue the electrolysis until deposition is essentially complete, as indicated by failure to plate on a new surface of the cathode stem, when the level of the solution is raised.

NOTE When a current of 1 A to 3 A is used, the electrolysis requires less than 10 h. Avoid extended electrolysis times.

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1) Platinum cathodes formed from plain or perforated sheets may also be used.

7.2.4 Without switching off the current, wash the cathode with a stream of water as it is being removed from the spent solution. Immediately wash successively in two baths of water and two baths of ethanol, methanol or other alcohols (4.6). Dry at 110 °C for 3 min to 5 min and cool to ambient temperature in a desiccator. Determine the mass of the metallic copper deposited.

Reserve the spent electrolyte.

7.2.5 Determine the residual copper in the spent electrolyte by a convenient spectrometric technique such as atomic absorption spectrometry or inductively coupled plasma optical emission spectrometry. The flame atomic absorption spectrometry method is described in the Annex A.

8 Expression of results

Calculate the copper mass fraction, in percent (%), using Formula (1):

$$w_{\mathrm{Cu}} = \frac{(m_1 - m_2) + m_3}{m_0} \times 100 \qquad (1)$$

where:

w_{Cu} is the copper mass fraction in per cent (%);

m_0 is the mass of the test portion (7.1), in grams (g);

m_1 is the mass of the cathode plus the electrodeposited copper, in grams (g);

m_2 is the mass of the cathode (5.2.1), in grams (g);

m_3 is the mass of residual copper found in the spent electrolyte (Annex A), in grams (g).

9 Precision

Twelve laboratories co-operated in testing this method and obtained the results summarised in Table 1.

Table 1 — Statistical information

Level	Reference value %	Found %	Repeatability r	Reproducibility R
1	99,89	99,837	0,074	0,106
2	≥ 99,99	99,988	0,013	0,032

NOTE Taking into account the narrowness of the scope (99,80 % to 100 %) and the discrepancy in the relation between the level of content and the r and R levels (r and R for the lowest level of content are higher than those for the highest level), the lg relationship between copper mass fraction (w_{Cu}) and r and R is irrelevant. Therefore, it is suitable to take 0,074 % and 0,106 % as the representative values for r and R respectively, for the whole scope of the method.

10 Test report

The test report shall contain the following information:

a) identification of the test sample;

b) reference to this European Standard (EN 16117-2);

c) method used;

d) results;

e) any unusual characteristics noted during the determination;

f) any operation not included in this European Standard or in the document to which reference is made or regarded as optional;

g) date of the test and/or date of preparation or signature of the test report;

h) signature of the responsible person.