SLOVENSKI STANDARD
SIST EN 13603:2004

01-januar-2004

Baker in bakrove zlitine ï Preskusne metode za ocenjevanje kakovosti zaġļ itnih kositrovih prevlek na okroglih bakrovih vleļ enih ģicah za elektrotehniko

Copper and copper alloys - Test methods for assessing protective tin coatings on drawn round copper wire for electrical purposes

Kupfer und Kupferlegierungen - Prüfverfahren zur Beurteilung von Schutzüberzügen aus Zinn auf gezogenen Runddrähten aus Kupfer für die Anwendung in der Elektrotechnik

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Cuivre et alliages de cuivre - Méthodes d'évaluation des revetements en étain sur les fils ronds étirés en cuivre pour usages électriques

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Ta slovenski standard je istoveten z: EN 13603:2002

ICS:

25.220.40	Kovinske prevleke	Metallic coatings
77.150.30	Bakreni izdelki	Copper products

SIST EN 13603:2004 en

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13603

March 2002

ICS 25.220.40; 77.150.30

English version

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This European Standard was approved by CEN on 22 February 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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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 Management Centre has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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SIST EN 13603:2004

Contents

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 13603:2004
https://standards.iteh.ai/catalog/standards/sist/32cc2dd9-757e-4580-a9b1-746353dc6eef/sist-en-13603-2004

SIST EN 13603:2004

Foreword

This document EN 13603:2002 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 September 2002, and conflicting national standards shall be withdrawn at the latest by September 2002.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 5 "Copper for electrical purposes" to prepare the following standard:

EN 13603, *Copper and copper alloys — Test methods for assessing protective tin coatings on drawn round copper wire for electrical purposes.*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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SIST EN 13603:2004
https://standards.iteh.ai/catalog/standards/sist/32cc2dd9-757e-4580-a9b1-746353dc6eef/sist-en-13603-2004

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Introduction

In preparing this document CEN/TC 133/WG 5 agreed to study carefully existing standards and to combine the best test methods for assessing tin coatings into a single European Standard.

The following standards were considered:

ISO 2177 : 1985;

ASTM B 33 : 1981, clauses 7 and 8;

BS 4109 : 1970, clause 14;

DIN 51213 : 1970, clause 6.

1 Scope

This European Standard specifies methods for assessing the tin coating on drawn round copper wire for the manufacture of electrical conductors, e.g. according to EN 13602.

Standard includes test methods for the determination of the following characteristics:

a) thickness of the unalloyed tin coating;

b) continuity of the tin coating;

c) adherence of the tin coating.

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WARNING This standard can involve the use of hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with their use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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https://standards.iteh.ai/catalog/standards/sist/32cc2dd9-757e-4580-a9b1-746353dc6eef/sist-en-13603-2004

2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 610, *Tin and tin alloys — Ingot tin.*

EN 13602, *Copper and copper alloys — Drawn, round copper wire for the manufacture of electrical conductors.*

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 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
unalloyed tin coating
layer of pure tin on the surface of tinned wire

SIST EN 13603:2004

3.2
alloyed tin coating
diffusion layer of copper and tin formed at the copper wire and tin coating interface during tinning and subsequent drawing and annealing processes

3.3
total tin coating
sum of the thicknesses of the unalloyed tin coating and the alloyed tin coating

3.4
measuring area
area of the surface over which a single measurement is made

4 Thickness of the unalloyed tin coating

4.1 Principle

Anodic dissolution of a well-defined area of the unalloyed coating using a suitable electrolyte, followed by detection of the virtually complete dissolution of the unalloyed coating by a rapid change in cell voltage. Calculation of the unalloyed coating thickness from the quantity of electricity (in coulombs) used, which can in turn be calculated from:

a) the time interval between the start of the test and the first rapid change of cell voltage, if it is conducted at constant current density; or

b) the integrated quantity of electricity used in dissolving the unalloyed coating.

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4.2 Reagents and materials

4.2.1 Electrolyte, either a hydrochloric acid electrolyte or an electrolyte recommended by the instrument manufacturer.

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For the hydrochloric acid electrolyte dilute 170 ml of hydrochloric acid (HCl), ϱ = 1,18 g/ml, to 1 000 ml with deionised water.

NOTE The unalloyed tin coating dissolves anodically at an efficiency of nearly 100 %; for determination of the electrolyte efficiency, see 4.5.6.

WARNING Hydrochloric acid causes burns and is irritating to the respiratory system. Avoid breathing the vapour and prevent contact with eyes and skin.

This electrolyte dissolves tin coatings at very low cell voltages at which there is no anodic attack on the substrates when they are exposed at the end of the test.

4.2.2 Tin, tin grade in accordance with EN 610.

4.3 Apparatus

4.3.1 General

Suitable instruments may be constructed from readily available components, alternatively a proprietary instrument may be used.

SIST EN 13603:2004

4.3.2 Direct reading instruments

Proprietary direct reading instruments are available for use with electrolytes recommended by the manufacturer.

The calculation of thickness of tin coating from current density is made electronically. The instrument shall have some means of indicating when the unalloyed tin coating has been fully removed.

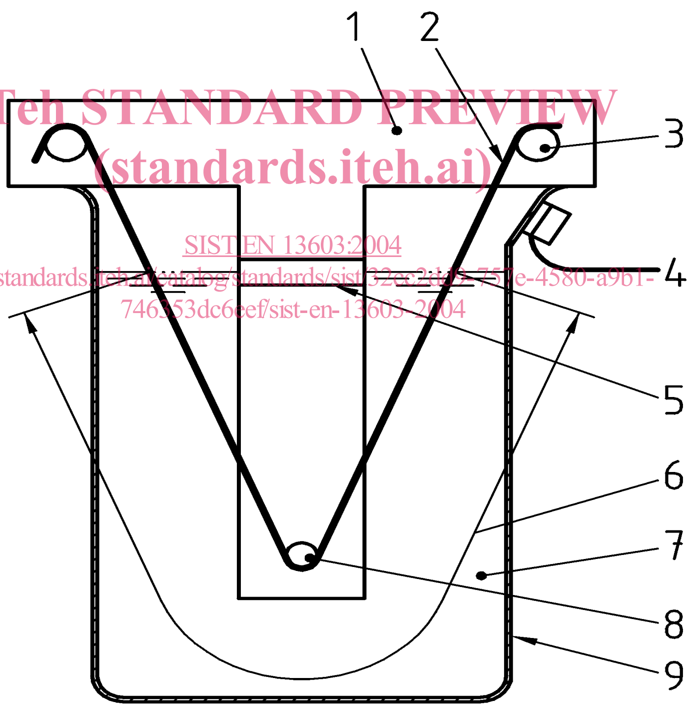
4.3.3 Other instruments

Instruments other than proprietary direct reading instruments record the quantity of electricity, in coulombs, used in dissolving the unalloyed coating from the measuring area, usually in arbitrary units, from which the thickness can be calculated using factors or tables.

4.3.4 Electrolytic cell

The electrolytic cell consists of a container for the electrolyte, a cathode and an anode which is the test sample. The container, if it is made of metal, for example stainless steel, can serve as the cathode. If the container is made of insulating material, a separate cathode is required.

Also required are a device for supporting the appropriate length of test sample and an agitation mechanism. Depending on the wire diameter, the test sample may be a straight length of wire or, if necessary to obtain sufficient surface area for smaller diameter wires, a holding device such as that shown schematically in Figure 1 is required. A magnetic stirrer or similar system shall be used to provide agitation.



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(standards.iteh.ai)
SIST EN 13603:2004

Key

1 Tee-frame to support test piece of non-conducting material, manufactured from nylon or other plastics
2 Test piece
3 Test piece connection (anode)
4 Cathode connection
5 Electrolyte level
6 Test piece length L
7 Electrolyte
8 Non-metallic pin
9 Cathode (stainless steel or lead), container (beaker)

Figure 1 — Alternative method for supporting fine wire or wire which cannot be straightened

SIST EN 13603:2004

4.4 Preparation of the test piece

Select a suitable length of test sample in order to provide the appropriate test piece area for exposure to the electrolyte. If necessary, clean the test surface with a suitable organic solvent (see 4.5.4.5).

NOTE Care should be taken to avoid removal of metal during the cleaning operation.

4.5 Procedure for determining the thickness of unalloyed tin coatings

4.5.1 General

If commercial equipment is used, follow the manufacturer's instructions with respect to the operating procedure for measurement, the electrolyte and, if necessary, calibration. Appropriate attention shall be given to the factors listed in 4.5.4. The performance of the instrument shall be checked using a reference specimen of pure tin wire. A tin grade in accordance with EN 610 shall be used. The test shall be carried out in accordance with 4.5.6.

If the instrument readings or the calculation of K give an electrolytic efficiency of equal to or greater than 98 %, the instrument may be used without further adjustment. Otherwise, the cause of discrepancy shall be remedied. Proprietary instruments shall be calibrated in accordance with the manufacturer's instructions.

4.5.2 Determination of measuring area

For the determination of the measuring area the length L of the test piece in millimetres shall be determined with an accuracy of 1 % and the diameter d of the test piece for wires with a nominal diameter of < 0,6 mm shall be determined with an accuracy of 1 % and for wires with a nominal diameter of ≥ 0,6 mm with an accuracy of 0,5 %. The measuring area A in square centimetres is given by the equation (1):

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$$A = \frac{d \times L \times \pi}{100} \tag{1}$$

NOTE An exact area of stripping is necessary for accuracy and the main source of error is due to the meniscus and current field at the electrolyte surface.

SIST EN 13603:2004
https://standards.iteh.ai/catalog/standards/sist/32cc2dd9-757e-4580-a9b1-746353dc6eef/sist-en-13603-2004

4.5.3 Electrolysis (Dissolution of the unalloyed tin coating)

The electrolyte (4.2.1) and test piece shall be introduced into the cell so that a known area is exposed to the electrolyte. Efforts shall be made to ensure that no gas bubbles occur on the measuring area by use of the agitation mechanism. The electrical connections shall be made and the agitator operated. Electrolysis shall be continued until dissolution of the unalloyed tin coating is complete, as indicated by a sharp change in the anode potential or cell voltage, or by the operation of the automatic cut-out.

After completion of the test, the test piece shall be removed from the cell, rinsed with water and examined to ensure that complete removal of the unalloyed tin coating has occurred over the measuring area, see 4.5.4.9.

4.5.4 Factors affecting the measuring accuracy

4.5.4.1 Coating thickness

The optimum accuracy is achieved with coating thicknesses in the range 0,2 µm up to 50 µm.

4.5.4.2 Current variation

For instruments using the constant current and time measuring technique, current variation will cause errors. For instruments using a current-time integrator, too large a change in current can change the anode current efficiency or interfere with the end-point, causing an error.