SLOVENSKI STANDARD
oSIST prEN 13603:2020

01-april-2020

Baker in bakrove zlitine - Preskusne metode za ocenjevanje kakovosti zaščitnih kositrovih prevlek na okroglih bakrovih vlečenih žicah 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

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

Ta slovenski standard je istoveten z: **prEN 13603 rev**

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25.220.40	Kovinske prevleke	Metallic coatings
77.150.30	Bakreni izdelki	Copper products

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 13603

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ICS 25.220.40; 77.150.30

Will supersede EN 13603:2013

English Version

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

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

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 133.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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prEN 13603:2020 (E)

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European foreword

This document (prEN 13603:2020) has been prepared by Technical Committee CEN/TC 133 "Copper and copper", the secretariat of which is held by DIN.

This document is currently submitted to the CEN enquiry.

This document will supersede EN 13603:2013.

In comparison with the previous edition, the following technical modifications have been made:

— Include the X-Ray fluorescence analysis (XRF) to measure the thickness of the tin layer.

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1 Scope

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

This document 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.

WARNING — This document can involve the use of hazardous materials, operations, and equipment. This document does not purport to address all of the safety problems associated with their use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Moreover this document does not cover the aspects related to the people protection against the X-ray. To obtain information applicable to this aspect it is convenient to refer to national and international standards, and also to the local regulations if they exist.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EN 610, *Tin and tin alloys - Ingot tin*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

• IEC Electropedia: available at http://www.electropedia.org/

• ISO Online browsing platform: available at http://www.iso.org/obp

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

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

3.5
X-Ray Fluorescence
XRF
secondary radiation occurring when a high intensity incident X-ray beam impinges upon a material placed in the path of the incident beam

Note 1 to entry: The secondary emission has wavelengths and energies characteristic of that material.

4 Thickness of the unalloyed tin coating

NOTE   This document refers to two different methods to measure the thickness of the tin coating layers on copper wires, the first one is based on the electrolytic dissolution of the tin layer. The second one is based on the direct measure of the tin layer on the wires by means of the reflection of an X-ray beam on its surface.

4.1 Principle of the method based on the electrolytic dissolution of the tin coating

4.1.1 General

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.

4.1.2 Reagents and materials

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

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.

4.1.2.2   Tin, tin grade in accordance with EN 610

4.1.3 Apparatus

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

4.1.3.1 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.

4.1.3.2 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.

4.1.3.3 Electrolytic cell

The electrolytic cell consists of a container for the electrolyte, a cathode and an anode, which is the test sample. If the container is made of metal, such as stainless steel, the container 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 the 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.
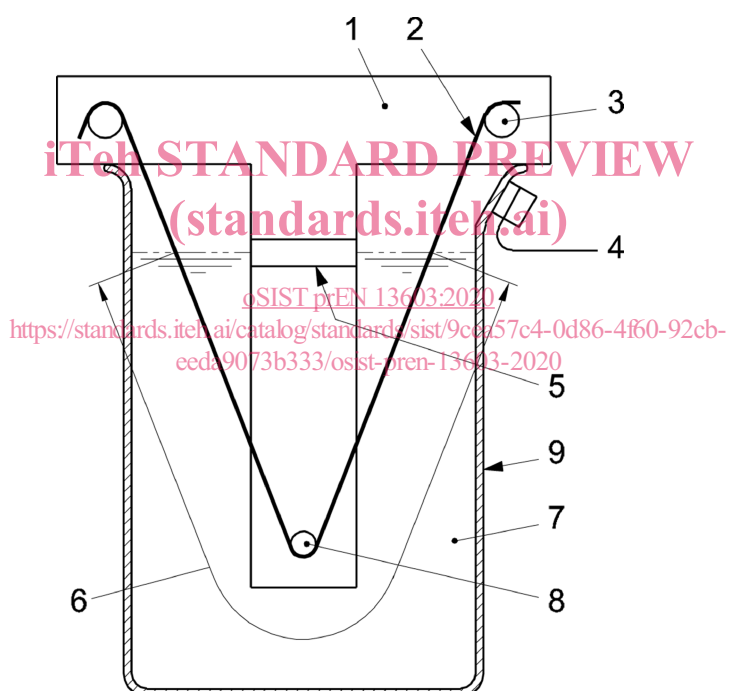
Key

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

4.1.4 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 5.5).

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

4.1.5 Procedure for determining the thickness of unalloyed tin coatings

4.1.5.1 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 4.1.5.4. 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 4.1.5.6.

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.

4.1.5.2 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 Formula (1):

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

(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.

4.1.5.3 Electrolysis (Dissolution of the unalloyed tin coating)

The electrolyte (4.1.2.1) 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 4.1.5.4.9).

4.1.5.4 Factors affecting the measuring accuracy

4.1.5.4.1 Coating thickness

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

4.1.5.4.2 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.

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4.1.5.4.3 Area variation

The accuracy of the thickness measurement will be no better than the accuracy to which the measuring area is known. Area variations due to electrolyte level (excessive agitation), can lead to measurement errors. In some cases it can be advantageous to measure the length after electrolysis is complete and re-calculate the area.

4.1.5.4.4 Agitation

Agitation (i.e. the rate of stirring) shall be sufficient to remove any gas bubbles formed during the test, which can adhere to the test piece or cathode. Excessive agitation shall be avoided to prevent interference with the length of test piece submerged.

4.1.5.4.5 Condition of the test piece surface

Oil, grease, paint, corrosion products, staining or other surface chemical treatments can interfere with the test.

4.1.5.4.6 Cleanliness of the cell

Deposition of tin can take place on the cathode in some electrolytes. This deposit can alter the cell voltages. It is, therefore, essential to keep the cathode clean.

4.1.5.4.7 Cleanliness of the electrical connections

In the case of instruments other than the constant current type, if the electrical connections are not clean, the current/potential relationship will be disturbed and false end-points obtained.

4.1.5.4.8 Calibration standards

Measurements made using calibration standards are subject to the additional error of the calibration standards.

4.1.5.4.9 Non-uniform dissolution

If the rate of dissolution is not uniform over the measuring area, a premature end-point can be obtained and yield low results. Examination of the surface shall be made after the test (see 4.1.5.3) to verify that most of the coating has dissolved.

The presence of other matter in the coating, the roughness of the coating surface and the presence of porosity in the coating can cause fluctuation of the cell voltage. Such fluctuation can affect the end-point.

4.1.5.4.10 Electrolyte efficiency

The determination of tin thickness by this method depends upon the efficiency of the electrolyte K being at least 98 %. The value of K should be determined periodically (see 4.1.5.6).

4.1.5.5 Measurement uncertainty

The test equipment and the procedure shall be such that the coating thickness can be measured to within a 5,0 % uncertainty under the following conditions:

- the electrical current shall be controlled within 10 mA;

- the time shall be controlled within 1 s;

- the area shall be controlled within the accuracy given in 4.5.2;

- the efficiency of the electrolyte K shall be greater than 98 % (see 4.1.5.6).

4.1.5.6 Determination of electrolyte efficiency

Where a manufacturer of apparatus recommends the use of a particular electrolyte for determining tin thickness on copper wire, the control of electrolyte efficiency shall be in accordance with the manufacturer's instructions. In other cases, the value of K shall be determined periodically using the following procedure:

a)   a suitable test anode shall be manufactured comprising wire in the range of diameter 0,5 mm up to and including 2,00 mm, of suitable length, coated with pure, unalloyed tin (4.1.2.2) to a thickness of at least 4 µm;

b)   the test anode shall be accurately weighed and its mass recorded;

c)   the test anode shall be immersed in the cell containing the electrolyte using either the same length as that recommended by the instrument manufacturer or used in the routine determination of tin thickness;

d)   the current shall be either that recommended by the instrument manufacturer or used in the routine determination of tin thickness;

e)   the duration of the test shall be sufficient to ensure a significant removal of tin but not so long that the tin coating is completely removed;

f)   the duration of the test in seconds and the current in amperes shall be noted;

g)   the anode shall be removed, washed, rinsed and dried without wiping;

h)   the anode shall be re-weighed to the same accuracy as before;

i)   the electrolytic efficiency K shall be calculated as follows:

$$K = \frac{m}{E \times I \times t}$$

(2)

where

   m is the loss in mass of anode, in milligram (mg);

   E is the electrochemical equivalent = 0,615 2 mg Sn/coulomb;

   I is the current, in amperes (A);

   t is the duration, in seconds (s).

In order to maintain precision in weighing, the ratio of the mass of the anode to the mass of tin removed should be 1 000 : 1.

NOTE   The value of 0,615 2 for electrochemical equivalent is based on the fact that 96 485 coulomb will remove 59,35 g of Sn^{2+} at 100 % efficiency.

If the value of K determined is less than 98 %, the electrolyte shall be discarded.