BS EN 13603:2013

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of EN 13603:2013. It supersedes BS EN 13603:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NFE/34/1, Wrought and unwrought copper and copper alloys.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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

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

This European Standard was approved by CEN on 25 April 2013.

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Foreword

This document (EN 13603:2013) has been prepared by Technical Committee CEN/TC 133 “Copper and copper”, 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of an identical text or by endorsement, at the latest by December 2013, and conflicting national standards shall be withdrawn at the latest by December 2013.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.

This document supersedes EN 13603:2002.

In comparison with EN 13603:2002, the following changes have been made:

— Editorial modifications.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 4 "Extruded and drawn products, forgings and scrap" to prepare the revision of the following standard:

EN 13603:2002, *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 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 methods for assessing the tin coating on drawn round copper wire for the manufacture of electrical conductors, e.g. according to EN 13602.

This European 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.

WARNING — This European 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.

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.

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

3 Terms and definitions

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

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 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.

4.2 Reagents and materials

4.2.1 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), $\rho = 1{,}18$ 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

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

4.3.1 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.

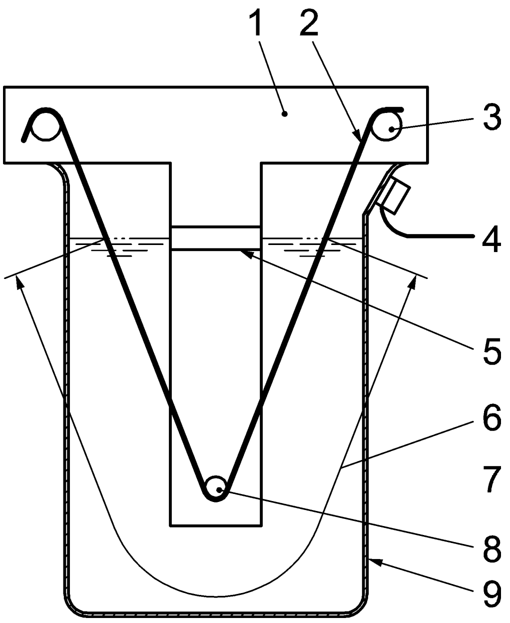
4.3.2 Other instruments

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.

4.3.3 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

4.4 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).

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 Formula (1):

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

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.

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.

4.5.4.3 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.

4.5.4.4 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.

4.5.4.5 Condition of the test piece surface

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

4.5.4.6 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.

4.5.4.7 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.

4.5.4.8 Calibration standards

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

4.5.4.9 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 4.5.3) 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.

4.5.4.10 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 4.5.6).

4.5.5 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 4.5.6).

4.5.6 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 (4.2.2.) 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:

$$K = \frac{m}{E \times I \times t} \qquad (2)$$

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^{++} at 100 % efficiency.

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

4.6 Expression of results

The unalloyed tin coating thickness tk, in micrometres, is given by the Formula (3):

$$tk = 100 \times K \times \frac{Q \times E}{A \times \rho} \quad \text{or} \quad 100 \times K \times \frac{I \times t \times E}{A \times \rho} \qquad (3)$$

where

K is the current efficiency of the dissolution process and shall be assumed to be 100 in the case of 100 % efficiency (see 4.5.6);

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

A is the area, in square centimetres (cm^2), from which the coating is dissolved, i.e. the measuring area;

ρ is the density, in grams per cubic centimetre (g/cm^3), of the unalloyed tin coating which for tin shall be 7,29 g/cm^3;

Q is the quantity of electricity, in coulomb, passed in dissolving the unalloyed tin coating; if an integrating meter is not used, calculate Q from Formula (4):

$$Q = I \times t \qquad (4)$$

where

I is the current, in amperes (A);

t is the test duration, in seconds (s).

Formula (3) above may be simplified, by substitution, to Formula (5):

$$tk = 0{,}008\,44 \times K \times \frac{Q}{A} \text{ i.e. } tk = 0{,}844 \times \frac{Q}{A} \qquad (5)$$

5 Continuity of the tin coating

5.1 Principle

A length of wire with a 5 000 mm^2 surface area shall be wrapped round a mandrel, cleaned and immersed in a test solution. Discolouration of the test solution, due to dissolution of copper, shall be compared to a reference solution of known copper concentration.

5.2 Test solution

Dissolve 10 g of chemically pure ammonium persulphate $[(NH_4)_2S_2O_8]$ in deionised water, add 20 ml of ammonia solution (NH_4OH), ρ = 0,88 g/ml up to 0,91 g/ml, and dilute to 1 000 ml.

NOTE The test solution cannot be stored because of loss of peroxide activity.

5.3 Reference solution

Dissolve 0,10 g of chemically pure copper sulphate ($CuSO_4 \cdot 5H_2O$) in deionised water, add 50 ml of ammonia solution (NH_4OH) ρ = 0,88 g/ml up to 0,91 g/ml and dilute to 1 000 ml. This solution has a copper concentration of 0,025 g/l.

5.4 Preparation of the test piece

A test piece length shall be permanently marked with a gauge length L, determined by reference to Table 1, such that there is 50 mm at either end of the gauge length for handling and support. Accuracy of marking shall be within 0,5 % L. The length L shall be wound on a smooth mandrel of the specified diameter (see Table 1), as an open helix. Care shall be used to avoid twisting or damage to the tinned coating. The 50 mm ends shall be positioned so that when tested, only the length L between the gauge marks will be immersed in the test solution.

Table 1 — Test parameters

Values in millimetres

Nominal wire diameter D		**Diameter of mandrel**	**Gauge length** L
over	up to and including		
0,04[a] 0,08 0,16	0,08 0,16 0,315	by agreement 15 ± 0,2 20 ± 0,2	[b]
0,315 0,5 1,0	0,5 1,0 1,5	25 ± 0,2 30 ± 0,2 40 ± 0,2	$L = \frac{1592}{D}$
1,5 3,0	3,0 5,0	50 ± 0,2 60 ± 0,2	

[a] Including 0,04.

[b] It shall be agreed between the purchaser and the supplier if wire with diameter from 0,04 mm up to and including 0,315 mm shall be tested. If agreed, it will also be necessary to agree test piece lengths and the pass/fail criteria in g/m^2 of copper dissolved (see 5.7). If a criterion different from that specified is agreed, it will also be necessary to determine and agree the copper concentration of the reference solution (see 5.3).

5.5 Cleaning of the test piece

Immediately before immersion in the test solution, the test piece shall be cleaned in a suitable volatile organic solvent. The solvent may be agitated but not the test piece. The test piece shall be dried before immersion in the test solution with hot air or wiped with a lint free cloth where appropriate.

5.6 Immersion for testing

Immediately after cleaning and drying, the test piece shall be immersed in 200 ml of the test solution such that only the gauge length L is immersed. The immersion time shall be 10 min. During the immersion time the solution shall not be agitated but maintained at a temperature of (20 ± 1) °C.

5.7 Determination

5.7.1 General

The colour density of the test solution shall be checked for acceptability by one of the methods given in 5.7.2 or 5.7.3.

5.7.2 Comparison by Nessler cylinders

A matched pair of 50 ml Nessler cylinders shall be used. One shall be filled with an undiluted volume of the test solution, the other with the same volume of reference solution. The density of colour of the two solutions in the Nessler cylinders shall be compared to a Nessleriser. The test solution shall not have a colour density greater than that of the reference solution. After determination the test solution shall be discarded.

NOTE The requirement specified in EN 13602 is max. 1,0 g of copper dissolved from 1 m^2 of wire. The test piece surface area is 5 000 mm^2, i.e. 0,005 m^2, i.e. 0,005 g of copper dissolved from 5 000 mm^2 o 1 g/m^2. If the 0,005 g of copper were dissolved in 200 ml of test solution, it would contain 0,025 g/l of copper.

The test solution and reference solution (see 5.3) are therefore directly comparable without dilution, calculation or use of colorimetric methods. The density of blue colour may be faint but compared with a Nessler cylinder of water the difference is clearly discernible.

5.7.3 Colorimetric method

Using suitable photometric apparatus, compare the optical density of the test solution with that of the reference solution (see 5.3). The test solution shall not have an optical density greater than that of the reference solution.

If a quantitative result is required, prepare a calibration graph by plotting optical density against percentage of copper using different concentrations of copper from a standard solution.

6 Adherence of the tin coating

6.1 Principle

A length of wire is twisted and wound round its own diameter or a mandrel as a close helix and immersed in a test solution to enhance any cracks which may occur. After removal from the solution, the wire is examined for evidence of black cracks.

6.2 Stock solution

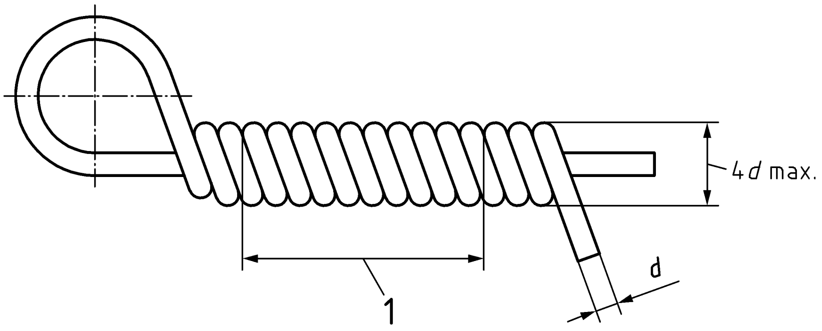
A stock solution of saturated sodium sulphide shall be prepared by dissolving commercial purity sodium monosulphide (Na_2S) in deionised water until the solution is saturated. To this solution sublimated sulphur (flowers of sulphur) shall be added in excess of 250 g/l. This solution shall be stored for 24 h before use.

6.3 Test solution

The test solution shall comprise a portion of the stock solution diluted with deionised water to ρ = 1,142 g/ml at (23 ± 2) °C.

6.4 Preparation of the test piece

A test piece of sufficient length shall be taken and 14 continuous turns wound around its own diameter (see Figure 2). Wires of less than 0,32 mm diameter may be wrapped around a smooth mandrel of a stated diameter agreed between the customer and the supplier.



Key

1 area to be examined (see 6.7)
d diameter of wire

Figure 2 — Preparation of the test piece

When the first two turns have been applied the wire remaining to be applied shall be subjected to 1½ permanently applied axial rotations. The remaining 12 turns shall then be applied. The force applied to the wire shall be sufficient to wind the helix. The overall diameter of the helically wrapped wire shall not exceed 4 d (see Figure 2).

6.5 Cleaning of the test piece

Immediately before immersion in the test solution, the test piece shall be cleaned in a suitable volatile organic solvent. The solvent may be agitated but not the test piece. The test piece shall be dried before immersion in the test solution with hot air or wiped with a lint free cloth where appropriate.

6.6 Immersion for testing

The wrapped test piece shall be fully immersed in the diluted sodium polysulfide solution ρ = 1,142 g/ml for a period of 30 s at a temperature between 16 °C and 21 °C. Neither the test piece nor the solution shall be agitated during the immersion period.

After 30 s the test piece shall be removed, washed with water and gently wiped dry to remove any deposits or grey colouration which may have occurred.

The test solution may be re-used until it fails to turn a piece of clean plain copper wire black within 5 s.

6.7 Examination

The whole exposed area of the ten turns in the middle of the wrapped test piece shall be examined (see Figure 2).

Cracks which have occurred during wrapping and where copper has been exposed will be black. Any other cracks, defects or faults or areas of clean copper or grey coloured areas shall be ignored.

The presence of any black crack shall indicate that the test piece has failed. Except for wires less than 0,32 mm diameter, samples shall be examined with the unaided eye corrected to normal vision if necessary. Wires less than 0,32 mm diameter may be examined at a magnification not exceeding × 5.

7 Test report

If a test report is required, it shall include the following information:

a) a reference to this European Standard (EN 13603);

b) which of the tests in accordance with this standard were carried out;

c) diameter of the wire tested in millimetres;

d) the origin of the samples, i.e. as tinned, tinned and drawn, tinned drawn and bunched, etc.;

e) the number of determinations and their results, in the case of:

    1) thickness: minimum, maximum and mean value in μm;

    2) continuity: passed/failed;

    3) adhesion: passed/failed;

f) any deviation from the methods specified or factors which may have influenced the results.

The test report shall be dated and signed.

Bibliography

[1] ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution*

[2] ASTM B33-10, *Standard specification for tin-coated soft or annealed copper wire for electrical purposes*

[3] EN 13602, *Copper and copper alloys — Drawn, round copper wire for the manufacture of electrical condoctors*

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