



Designation: B1019 – 21

Standard Test Method for Determination of Surface Oxides on Copper Rod (for Electrical Purposes)¹

This standard is issued under the fixed designation B1019; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification established a quantitative, laboratory conducted, electrolytic reduction method to determine the thickness of surface copper oxide films on copper rod produced to Specification B49 for further fabrication into electrical conductors.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 The following documents in the current issue of the *Annual Book of ASTM Standards* form a part of this specification to the extent referenced herein and define materials suitable for use in rod manufacture.

2.2 *ASTM Standards*:²

B49 Specification for Copper Rod for Electrical Purposes

B846 Terminology for Copper and Copper Alloys

D1193 Specification for Reagent Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

¹ This test method is under the jurisdiction of ASTM Committee B05 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.06 on Methods of Test.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E2480 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method with Multi-Valued Measurands

E2653 Practice for Conducting an Interlaboratory Study to Determine Precision Estimates for a Fire Test Method with Fewer Than Six Participating Laboratories

3. Terminology

3.1 For definitions of related terms to copper and copper alloys, refer to Terminology B846.

4. Summary of Test Method

4.1 The thickness and type of unreduced oxide films remaining on the surface of copper rod after cleaning shall be determined by an electrolytic reduction method. This test shall be performed by reducing the surface copper oxide(s) to copper in an electrolytic cell. For a description of a similar, yet alternative standard procedure to determine tarnish films on coupons exposed to environmental tests, see Refs. (1-5).³ As shown by the schematic diagram in Fig. 1, the test sample being tested acts as cathode with respect to an anode, which shall be made from a platinum wire or an equivalent inert electrode. Current shall be supplied from a DC power supply or a coulometer. A discussion on means to help improve accuracy and repeatability of this test method will be found in Note X1.1.

4.2 Each of the copper oxides found on copper, namely cuprous and cupric, are reduced sequentially to copper at different reduction potentials, and the voltages are to be recorded against time during the entire test. When the individual reactions between the oxides and hydrogen ions are complete, gaseous hydrogen is generated and may be seen as little bubbles at the surface of the copper rod sample.

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

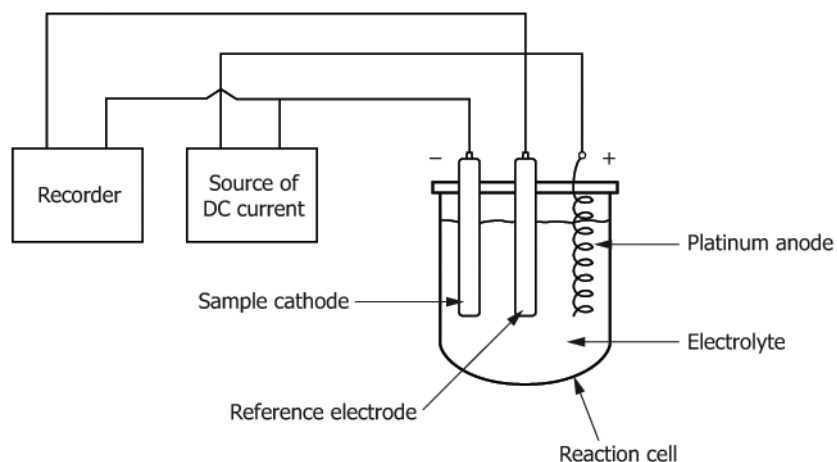


FIG. 1 Schematic Illustration Showing Electrolytic Reduction Test Method

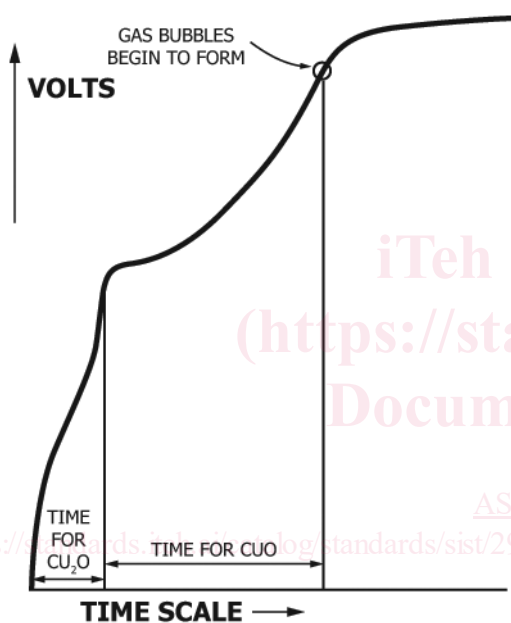


FIG. 2 Typical Voltage-Time Curve for Reduction of Copper Oxide Films

4.3 A typical curve of voltage versus time is presented in Fig. 2. Cuprous oxide is reduced initially. When this reaction is complete, reduction of the cupric oxide occurs at a higher voltage.

5. Significance and Use

5.1 The copper oxides layer present on the surface of the copper rod has a detrimental effect on both final surface quality of the copper wire produced by cold drawing of the rod, and on the drawing process itself leading to wire breaks and excessive wear of the drawing dies. Thus, it is critical to use adequate cleaning techniques during the copper rod manufacturing process in order to limit the depth of the residual copper oxide layer on the finished rod, and it is necessary to have a method to accurately measure the copper oxide depth.

5.2 This test method is the most common test method used by continuous casting and rolling mills to measure the depth of residual surface oxides after the copper rod has been cleaned.

5.3 Applicability of the test method for any other purpose or product has not been evaluated. Suitability beyond the stated scope shall be evaluated before use.

6. Interferences

6.1 *Sample Cleanliness*—The copper rod sample shall be free of any contamination following handling (as finger prints, tarnish, or oxidized surface); any residue on the sample, including mill, quench solution, or corrosion inhibitor film, shall be reasonably cleaned to prevent contamination of the electrolyte. This procedure is particularly important if the rod is acid-cleaned since the test is very sensitive to pH.

6.2 The test sample is cleaned by wiping with a cloth moistened with standard ACS grade acetone at room temperature.

6.3 *Test Delay*—The experience showed that visible surface tarnish or copper oxides could not generate the same test results of copper oxide film thickness when just made. For a representative result, the copper rod shall be sampled during production, stored in laboratory conditions, and the test shall be performed within a reasonable timeframe (within a few days from the production of the copper rod).

6.4 Electrolyte Quality:

6.4.1 Electrolyte quality has a significant effect on the test results, inasmuch as contamination or depleted electrolyte tends to reduce the reduction efficiency.

6.4.2 The electrolyte solution shall be replaced when testing of foil standards show a change of readings or when turning “cloudy.” The electrolyte is typically changed weekly.

6.5 *Dissolved Oxygen in Electrolyte*—Since oxygen is also introduced into the electrolyte at the end of the test when hydrogen gas is generated by electrolysis, the test is considered completed when bubbles are observed at the cathode (copper rod sample).

7. Apparatus

7.1 Fig. 1 shows the schematic illustration of the apparatus and includes:

7.1.1 A reaction cell, typically a glass beaker of 2000 mL volume.

7.1.2 A source of DC current, or a coulometer operating in the range of 1 mA to 20 mA.

7.1.2.1 *Current Density*—This property is calculated by taking the constant test current and dividing it by the surface area of the sample exposed to the electrolyte. The test takes more time to complete as the current density is decreased, but at the same time accuracy and repeatability are improved. As a compromise between attaining practical (short) laboratory test times while not losing extreme accuracy, a current density in the range between 0.15 mA/cm² and 0.55 mA/cm² is used in the rod industry.

7.1.3 A Platinum anode, or an equivalent inert electrode.

7.1.4 A reference electrode:

7.1.4.1 Either saturated calomel, or a silver/silver-chloride configuration can be used to determine voltage. However, if the current density is maintained fairly low, a simple copper wire can be used as an electrode with no effect on accuracy of the oxide thickness determination.

7.1.5 A recorder measuring the voltage between the sample (cathode) and the reference electrode. Range covering 0 V to 2 V.

7.1.6 A sample cathode consisting of a straight length of copper rod cut to a suitable length and suspended such that a known surface area of the rod is immersed in the electrolyte.

7.1.7 Connectors and wires to connect the DC current source and the recorder to the cathode, anode, and reference electrode.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type grade III of Specification **D1193**.

8.3 *Electrolyte Solution*—A 0.1 molar solution of sodium carbonate has generally been adopted, although potassium chloride solutions are also acceptable.

9. Hazards

9.1 No exceptional hazards are associated with this test method.

9.2 Users shall use normal laboratory safety measures and personal safety equipment.

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

10. Sampling, Test Specimens, and Test Units

10.1 Any rod diameter (or wire diameter) can be used. However, for consistent results, the minimum wire diameter shall be around 2 mm. Oxide thicknesses are reported in “Angstrom” units.

11. Preparation of Apparatus

11.1 Apparatus is arranged and connected as shown in **Fig. 1**.

12. Calibration and Standardization

12.1 No specific standards exist for this test method.

12.2 DC source and voltage recorder shall be checked and calibrated periodically.

13. Conditioning

13.1 No conditioning is required.

14. Procedure

14.1 The following is the method to be used:

14.1.1 A 30 cm length of rod is cut from a rod coil. The rod is straightened, cleaned with acetone, and marked to indicate the area to be submerged in the electrolyte.

14.1.2 For 8 mm diameter rod, the depth of submersion is 10 cm.

14.1.3 The sample is immersed in the electrolyte up to the mark and connected as in **Fig. 1**.

14.1.4 The tester is initiated until the voltage asymptote is reached, then the test is stopped.

14.1.5 The time taken to complete each reaction is measured.

14.1.6 The thickness of each oxide is calculated from **Eq 1**.

14.1.7 The test is conducted at room temperature.

14.1.8 The current of the DC source is set at 10 mA for the duration of the test.

15. Calculation or Interpretation of Results

15.1 Each of the copper oxides found on copper, namely cuprous and cupric, are reduced sequentially to copper at different reduction potentials, and the voltages are to be recorded against time during the entire test. When the individual reactions between the oxides and hydrogen ions are complete, gaseous hydrogen is evolved and may be seen visually at the surface of the copper rod sample.

15.2 A typical curve of voltage versus time is presented in **Fig. 2**. Cuprous oxide is reduced initially. When this reaction is complete, reduction of the cupric oxide occurs at a higher voltage.

15.3 Thickness of each oxide present shall be calculated as follows:

$$T = \frac{ItM}{SdFn} \quad (1)$$