



Standard Test Method for Calibration of Atmospheric Corrosion Test Chambers by Change in Mass of Copper Coupons¹

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1. Scope

1.1 This test method covers the calibration of atmospheric corrosion test chambers for electrical contacts that produce an adherent film of corrosion product on copper, such as a test comprised of a mixture of flowing gases that react with copper.

1.2 This test method is not applicable to tests where corrosion products may be removed from a copper surface during the test by fluids.

1.3 This test method is not applicable to tests where airborne solid or liquid material may be deposited on a copper surface during the test, as in a test which includes particulates suspended in the atmosphere.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The values stated in SI units are to be regarded as the standard.

2. Summary of Test Method

2.1 Copper coupon samples of a well-defined size are prepared and each is labeled for identification. All such coupons are cleaned by a standard process. Each coupon is weighed and the value is recorded. All coupons are exposed to the corrosion test for a specified time. The coupons are removed from the test and weighed and the new values are recorded. The change in weight for each coupon is calculated. The coupons are subjected to additional analysis as appropriate to determine the composition and thickness of any films present on the surface.

3. Significance and Use

3.1 Electrical devices that contain electrical contacts generally contain some copper-based materials. Atmospheric corrosion of copper parts in such devices often occurs in service environments. A quantitative measure of the effect of a laboratory corrosion test on copper permits assessment of the severity of the test. In addition, corrosion tests may be defined

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in terms of their effect on copper; this test method provides a way of comparing one test against a standard defined elsewhere, or allows a comparison of the performance of a test over a period of time. Although this test method provides for a relatively simple check of a test, the user is advised that additional analysis of the test chamber ambient is generally required to reproduce test conditions.

3.2 Atmospheric corrosion tests are used on a variety of materials besides copper. Care should be exercised in drawing conclusions about the effects on such materials of apparently equivalent tests if the composition of gases or experimental conditions are different. The primary use of this calibration test method is to assure correlation among nominally identical tests.

4. Apparatus

4.1 *Racks or Fixtures* suitable for holding the copper coupons in the test chamber are required. Make these fixtures from a material that is not attacked by the corrosion test. The fixtures shall be designed to:

4.1.1 Hold the coupons in a vertical orientation,

4.1.2 Hold the coupons so that they do not show any perceptible motion when observed with the unaided eye during the test,

4.1.3 Cover less than 5 % of the entire coupon surface area,

4.1.4 Touch the coupon only with electrically insulating parts and,

4.1.5 Allow free circulation of the ambient on both sides of the coupon.

4.2 In general, design all parts of the fixture to permit maximum circulation of the ambient around the coupon surfaces. The size and positioning of the holding fixtures depends on the chamber size. Section 6 of this test method gives requirements on the number and spacing of coupons; the holding fixtures must be designed to comply with these requirements.

4.3 *Balance*, with a capacity of at least 2 g and a resolution of 5 μg is required. Maintain the ambient in the vicinity of the balance between 20 and 50 % relative humidity.

4.4 *Fume Hood*, to conduct the chemical cleaning procedure of the coupons is required.

5. Reagents and Materials

5.1 *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 Society² where such specifications are available. 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.

6. Procedure

6.1 Use the requirements listed in 6.1.1 through 6.1.3 to determine the number of copper coupon specimens to be prepared.

6.1.1 For chambers with cubic working volumes, use the length of an edge to determine the number and placement of coupons in accordance with the following rules. For chambers with an edge dimension of 0.9 m or less, construct a reference grid with three equally spaced lines in each direction with the outer lines 0.1 m from the chamber wall and the third line centered between the outer lines. Place a coupon at each intersection of grid lines (27 coupons required). For chambers with an edge dimension of 0.9 m to 1.5 m, construct a reference grid with four equally spaced lines in each direction. The outer lines are placed 0.1 m from the chamber wall. Place a coupon at each intersection of grid lines (64 coupons required). For chambers with a maximum edge dimension greater than 1.5 m but less than 2.0 m, construct a reference grid with five equally spaced lines in each direction. The outer lines are placed 0.1 m from the chamber wall. Place a coupon at each intersection of grid lines (125 coupons required).

6.1.2 For rectangular chambers with non-cubic working volumes, the guidelines for each dimension of the chamber along the *x*, *y*, and *z* axes of the chamber shall follow the appropriate guidelines applied to cubic chambers. Thus, if the *x* and *z* axes are 1.4 m and the *y* axis is 0.8 m, four grid lines would be used for the *x* and *z* axes and 3 grid lines would be used for the *y* axis when determining the grid for coupon location.

6.1.3 For chambers with working volumes of other shapes than those covered in the preceding sections, devise a logical placement pattern with a coupon density roughly equal to that specified for rectangular chambers.

6.2 Select an appropriate duration of exposure for the copper coupons. Base this selection primarily upon the time required for the test ambient to produce a statistically significant weight change. Additional exposure time may be added to comply with other applicable requirements, specifications, agreements, etc., or for the convenience of the test operators, or both. Generally, the test time shall be one or more whole days with a tolerance of ± 1 h.

6.3 Prepare the test coupons from wrought, annealed, oxygen-free copper (99.95 % copper minimum, copper alloy C10200) sheet. Select a thickness of sheet sufficiently stiff to

resist bending during handling during the test but not so thick that the edges become a significant portion of the surface area. In general, thicknesses between 0.1 and 0.6 mm are recommended. Obtain material with a surface roughness less than 0.15 μm center line average and use the material with the as-rolled surface finish. Make the coupons squares or rectangles preferably 12.5 ± 1.2 mm wide. One or two holes 2.5 mm or less in diameter may be added to aid in mounting. Inspect the edges of the coupons at 10 \times magnification for the presence of burrs or slivers. If such features are found, they must be removed since they may corrode at a much higher rate than the coupon surface.

6.4 Mark all coupons with a code giving each coupon a unique identification. Make the characters in the code marking about 2 mm high by engraving or stamping without ink.

6.5 During the cleaning procedure and at all times after cleaning, handle coupons only with clean tweezers grasping in the region around the identification marking. Place each copper coupon in a separate clean, dry glass vial of an appropriate size such that only the edges and corners of the coupons touch the glass surfaces.

6.6 Clean all coupons in accordance with the procedure given in 6.6.1 through 6.6.7. Where a liquid bath is required, fill an appropriate vessel to a depth equal to or greater than 25 mm plus the largest dimension of the coupon sample to be cleaned. Unless otherwise directed, change the fluid in all baths after 50 coupons have been processed. Unless otherwise directed, use all baths at room temperature.

NOTE 1—**Precaution**—Conduct all operations involving acids and solvents in a fume hood.

NOTE 2—**Precaution**—Dispose of all acids and solvents in a safe, legally acceptable manner.

NOTE 3—The goal of 6.6.1 through 6.6.2 is to produce a coupon surface reasonably free of oils, greases, particulate debris and similar contaminants. Alternative test methods which achieve the same result are acceptable. Such alternative test methods may be necessary or desirable depending upon local conditions or facilities.

6.6.1 Immerse a coupon or small group of coupons for 5 to 10 minutes in an ultrasonic cleaner containing hot (65–85°C) 2% aqueous solution of a mildly alkaline (pH7.5–10) detergent.

6.6.2 Remove an individual coupon from the cleaner and rinse under warm running tap water for 5–15 seconds. Transfer to the next step immediately.

6.6.3 Transfer the coupon into a solution of 1 part concentrated hydrochloric acid and 3 parts deionized water; suspend coupon vertically for 120 ± 10 s in this solution.

6.6.4 Transfer the coupon immediately without allowing drying of surface into one of the following treatments: suspend coupon vertically in flowing deionized water for 15 ± 5 s or, suspend coupon with agitation for 15 ± 5 s in one vessel of deionized water, then immediately repeat in a second vessel of fresh deionized water.

6.6.5 Transfer the coupon immediately without allowing drying of surface to a bath of methanol; suspend the coupon vertically for 30 to 60 s with agitation in the bath.

6.6.6 Remove the coupon from methanol, immediately blow dry with prepurified nitrogen or with clean, dry air.

6.6.7 Place the coupon immediately in its glass vial, leave

² *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.