Designation: B810 – 01a (Reapproved 2017)

Standard Test Method for Calibration of Atmospheric Corrosion Test Chambers by Change in Mass of Copper Coupons[1]

This standard is issued under the fixed designation B810; 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 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 become familiar with all hazards including those identified in the appropriate Safety Data Sheet (SDS) for this product/material as provided by the manufacturer, to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.*

1.6 *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 *ASTM Standards:*[2]

B845 Guide for Mixed Flowing Gas (MFG) Tests for Electrical Contacts

3. Summary of Test Method

3.1 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.

4. Significance and Use

4.1 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 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.

4.2 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.

[1] This test method is under the jurisdiction of ASTM Committee B02 on Nonferrous Metals and Alloys and is the direct responsibility of Subcommittee B02.11 on Electrical Contact Test Methods.

Current edition approved April 1, 2017. Published April 2017. Originally approved in 1991. Last previous edition approved in 2011 as B810 – 01a (2011). DOI: 10.1520/B0810-01AR17.

[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.

5. Apparatus

5.1 *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:

5.1.1 Hold the coupons in a vertical orientation,

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

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

5.1.4 Touch the coupon only with electrically insulating parts and,

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

5.2 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 7 of this test method gives requirements on the number and spacing of coupons; the holding fixtures must be designed to comply with these requirements.

5.3 *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.

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

6. Reagents and Materials

6.1 *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 [3] 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.

7. Procedure

7.1 Use the requirements listed in 7.1.1 through 7.1.3 to determine the number of copper coupon specimens to be prepared.

7.1.1 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 1.4 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 1.4 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).

7.1.2 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 1.5 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.

7.1.3 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.

7.2 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.

7.3 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× 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.

7.4 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.

7.5 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.

7.6 Clean all coupons in accordance with either of the two procedures given in 7.6.1 through 7.6.2.5. 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

[3] *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.

otherwise directed, use all baths at room temperature. (**Warning—**Conduct all operations involving acids and solvents in a fume hood. Please be careful of N-Hexane. It is extremely flammable.)(**Warning—**Dispose of all acids and solvents in a safe, legally acceptable manner.)

NOTE 1—The goal of Cleaning Methods 1 and 2 are to produce a coupon surface reasonably free of oils, greases, particulate debris and oxides. Alternative cleaning methods which achieve the same result are acceptable. See the Appendix X1 for an example of comparison cleaning method research. Such alternative cleaning methods may be necessary or desirable depending upon local conditions or facilities.

7.6.1 *Cleaning Method 1:*

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

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

7.6.1.3 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.

7.6.1.4 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.

7.6.1.5 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.

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

7.6.1.7 Place the coupon immediately in its glass vial, leave vial uncapped for 10 min, then cap vial unless proceeding immediately to the next step.

7.6.2 *Cleaning Method 2:*

7.6.2.1 Immerse the coupon to be cleaned in n-Hexane for 120 s. Allow to drip dry for 10 s.

7.6.2.2 Immerse the coupon in Alphametals Lonco flux 3355–11 or equivalent material for 15 s. Allow to drip dry for 10 s.

7.6.2.3 Rinse the coupon twice with deionized water for 15 s.

7.6.2.4 Rinse coupon in methanol for 15 s and then allow to dry.

7.6.2.5 Place the coupon immediately in its glass vial, leave vial uncapped for 10 min, then cap vial unless proceeding immediately to the next step.

7.7 Allow all copper coupons to equilibrate in the vicinity of the balance for at least 30 min by placing the vials containing the coupons near the balance and removing the cap from each vial. Also, wait long enough so that at least 60 min has elapsed from the time that cleaning procedure was completed until the weighing begins. Weigh each coupon individually, replacing each in its glass vial after weighing. Reweigh two of the coupons to check for reproducibility. If the second weight reading does not differ by more than 1.0 μg/cm^2 of exposed surface area, proceed to the next step. If the readings differ by more than the preceding requirement, reweigh all coupons and repeat as required until stable weights are obtained.

7.8 Immediately install all coupons on holding fixtures in the corrosion test chamber. Record the time that the coupons are placed into the corrosion test.

7.9 After the appropriate test time for the test method in use has elapsed, remove the coupons from the holding fixture, placing each in an uncapped vial. Place the vials containing the coupons in the vicinity of the balance and wait at least 30 min to allow the coupons to come to equilibrium with room temperature and humidity.

7.10 Weigh all coupons, reweigh each coupon as required until a stable reading is obtained for each coupon.

7.11 Calculate the difference in weight before and after exposure to the corrosion test.

8. Report

8.1 Report the results in the data report format shown in Fig. 1.

9. Precision and Bias

9.1 *Precision*—An interlaboratory round robin involving six laboratories produced the following results. Each laboratory prepared 8 or 12 coupons by the procedures specified in this standard. All coupons from all laboratories were exposed simultaneously for 10 days to a Guide B845 Method G test environment. The mass gain on each coupon (exposed area of 7.8 cm^2) was measured, Table 1 summarizes the results. The results show that the minimum mass change within a laboratory ranges from 56 to 71 percent of the maximum mass

1. Date test started:
2. Date test ended:
3. Duration of exposure, that is, hours or days in the test chamber.
4. Test operator(s):
5. Designation of test chamber in which coupons were exposed:
6. Dimensions of test coupons (nominal dimensions may be reported provided that the actual dimensions do not differ by more than 2 % from nominal):
7. Test conditions, for example, gas concentrations, temperature, relative humidity, air flow rates in chamber, etc.:
8. State what deviations from the procedure specified in this standard were made or occurred (if none, so state):
9. Attach a map or comparable documentation that relates the identification number of each sample to the position of that sample in the test chamber.

Test Results

Identification Number of Sample	Exposed Area of Sample[A]	Initial Mass	Final Mass	Mass Change

[A] Record total area of all exposed coupon surfaces.

FIG. 1 Sample Data Report Form

TABLE 1 Results of Round Robin on Copper Coupon Mass Gain Conducted by Six Laboratories

	Laboratory ID					
	1	2	3	4	5	6
Minimum mass gain (mg)	0.2874	0.2746	0.2512	0.3020	0.3131	0.2995
Maximum mass gain (mg)	0.4759	0.4410	0.4455	0.4514	0.4417	0.4760
Mean mass gain (mg)	0.3870	0.3858	0.3639	0.3823	0.3840	0.3925
Median mass gain (mg)	0.3936	0.3939	0.3764	0.3939	0.3934	0.4006
Standard deviation of mass gain (mg)	0.0499	0.0464	0.0636	0.0508	0.0386	0.0619
Number of coupons	12	12	12	12	12	8

change for that laboratory, while, in comparing the mean mass changes for the six laboratories, the minimum is 93 percent of the maximum. Based on analysis of trends in mass gain with location, it appears that some of the difference in mass gain between coupons from a single laboratory came from differences in corrosion rate in different locations within the test chamber.

9.2 *Bias*—The procedure in this test method for calibrating atmospheric corrosion tests has no bias because the value of the weight change of a copper coupon in a given atmospheric corrosion test can be defined only in terms of a test method.

10. Keywords

10.1 accelerated testing; atmospheric corrosion; cleaning technique; contacts; copper; electrical contacts; mass change; mixed flowing gas testing; test calibration; weight gain

APPENDIX

(Nonmandatory Information)

X1. TYPICAL MASS CHANGE VALUES FOR COPPER COUPONS EXPOSED IN SOME MFG TESTS

X1.1 Table X1.1 lists mass change values measured on copper coupons in tests conducted by several organizations in 11 different test chambers. These values may be used as a general guide to the magnitude of changes to be anticipated in the various tests, but should not be used as the basis of test requirements since they are not part of a controlled laboratory round robin. Each mass change value is the average of the values from several coupons exposed simultaneously in the chamber during the test, typically 3 to 12 coupons.

TABLE X1.1 Typical Mass Change Values for Copper Coupons Exposed in Some MFG Tests

Guide B845 Test Method	Coupon Exposure Duration (days)	Mass Change per Day (mg/cm^2·day)
E	6.8	0.064
E	10	0.057
E	10	0.055
E	10	0.063
E	10	0.061
E	10	0.057
E	10	0.065
E	10	0.040
E	10	0.033
E	10	0.037
E	5	0.060
E	10	0.057
E	10	0.046
G	10.8	0.057
G	14	0.014
G	14.25	0.004
G	10	0.004
G	15	0.010
G	10	0.014
G	10	0.013
G	10	0.013
G	10	0.015
G	10	0.015
G	10	0.008
G	10	0.012
G	10	0.006
G	10	0.009
G	10	0.020
G	10	0.016
G	4	0.015
H	10	0.033
H	10	0.035
H	10	0.032
H	10	0.040
H + 200ppbSO2	10	0.045
H + 200ppbSO2	2	0.033
H + 200ppbSO2	4	0.029
H + 200ppbSO2	2	0.022
H + 200ppbSO2	2	0.025
N	10	0.022
N	10	0.023
N	10	0.018
N	10	0.022
N	10	0.037
N	10	0.014
N	10	0.013
N	10	0.019
N	10	0.016
N	10	0.007
N	10	0.019
N	10	0.012
N	10	0.012
N	10	0.026
N	10	0.014
N	10	0.013
N	5	0.014
N	5	0.013