



Designation: B 809 – 95 (Reapproved 2003)

Standard Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers-of-Sulfur”)¹

This standard is issued under the fixed designation B 809; 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 standard covers equipment and test methods for determining the porosity of metallic coatings, where the pores penetrate down to a silver, copper, or copper-alloy substrate.

1.2 This test method is suitable for coatings consisting of single or combined layers of any coating that does not significantly tarnish in a reduced sulfur atmosphere, such as gold, nickel, tin, tin-lead, and palladium, or their alloys.

1.3 This test method is designed to determine whether the porosity level is less than or greater than some value which by experience is considered by the user to be acceptable for the intended application.

1.4 Recent reviews of porosity testing and testing methods can be found in the literature.^{2,3} Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits and related metallic coatings. Other porosity test standards are Test Methods B 735, B 741, B 798, and B 799.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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.* For specific hazards statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:

B 374 Terminology Relating to Electroplating⁴

B 542 Terminology Relating to Electrical Contacts and Their Use⁵

B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor⁵

B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography⁵

B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings⁴

B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography⁵

B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor⁵

3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminologies B 374 and B 542.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *corrosion products*—reaction products of the basis metal or underplate, that protrude from, or are otherwise attached to, the coating surface after the test exposure.

3.2.2 *measurement area*—in this test method, that portion or portions of the surface that is examined for the presence of porosity. The measurement area shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

3.2.3 *metallic coatings*—in this test method, include platings, claddings, or other metallic coatings applied to the substrate. The coating can comprise a single metallic layer or a combination of metallic layers.

3.2.4 *porosity*—the presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal (see Guide B 765).

3.2.5 *significant surface*—of a coated part, is that portion (or portions) of the coating surface that is essential to the serviceability or function of the part, or which can be the source of corrosion products or tarnish films that interfere with the function of the part. For many plated products, the critical surface is identical to the measurement area.

3.2.6 *tarnish*—reaction products of copper or silver with oxygen or reduced sulfur (that is, hydrogen sulfide (H₂S) and elemental sulfur vapor, but not sulfur dioxide (SO₂) or other sulfur oxides). They consist of thin films or spots that do not protrude significantly from the surface of the metallic coating (in contrast to *corrosion products*).

¹ This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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² Clarke, M., “Porosity and Porosity Tests,” *Properties of Electrodeposits*, Sard, Leidheiser, and Ogburn, eds., The Electrochemical Society, 1975, p. 122.

³ Krumbain, S. J., “Porosity Testing of Contact Platings,” *Transactions of the Connectors and Interconnection Technology Symposium*, Philadelphia, PA, October 1987, p. 47.

⁴ *Annual Book of ASTM Standards*, Vol 02.05.

⁵ *Annual Book of ASTM Standards*, Vol 02.04.



3.2.7 *tarnish creepage*—movement of tarnish films across the surface of the coating, the tarnish having originated either from pores or cracks in the coating or from areas of bare silver, copper, or copper alloy near the measurement area (as in a cut edge).

3.2.8 *underplate(s)*—a metallic coating layer(s) between the substrate and the topmost layer or layers. The thickness of an underplate is usually greater than 1 μm (40 $\mu\text{in.}$).

4. Summary of Test Method

4.1 The test specimens are suspended over “flowers-of-sulfur” (powdered sulfur) in a vented container at controlled elevated relative humidity and temperature. Elemental sulfur vapor, which always exists in equilibrium with sulfur powder in a closed system, attacks any exposed silver, copper, or copper alloy, such as at the bottom of pores. Brown or black tarnish spots indicate porosity.

4.2 Exposure periods may vary, depending on the extent of porosity to be revealed.

4.3 This test involves tarnish or oxidation (corrosion) reactions in which the products delineate defect sites in coatings. Since the chemistry and properties of these products may not resemble those found in natural or service environments, this test is not recommended for prediction of product performance unless correlation is first established with service experience (but see 5.3).

5. Significance and Use

5.1 A major use of this test procedure is for determining coating quality. Porosity tests are indications of the completeness of protection or coverage offered by the coatings, since the coatings described in 1.2 are intended to be protective when properly applied. The porosity test results are therefore a measure of the deposition process control.

5.2 A particular purpose of the humid sulfur vapor test is for determining the quality of underplates of nickel or nickel alloy in those finish systems that have thin, 1.2 μm or less (50 $\mu\text{in.}$ or less) top layers above the nickel, since porosity in the underplate usually continues into such top layers.

5.3 The humid sulfur vapor test is often used as an environmental test to simulate many indoor humid atmosphere tarnishing and tarnish creepage effects. However, the chemistry and properties of these tarnish films may not resemble those found in other service environments. For such product performance evaluations, the test should only be used in combination with other performance evaluation tests, as specified in the referencing document for that product.

5.4 Porosity tests differ from corrosion and aging tests, since the latter are intended to measure the chemical inertness of the coating. In contrast, in a good porosity test procedure the corrosive agent should not attack the coating. It must instead, clean, depolarize, or activate the substrate metal exposed by the pore, or both, and attack it sufficiently to cause reaction products to fill the pore to the surface of the coating.

5.5 The humid sulfur test is highly sensitive, and is capable of detecting virtually all porosity that penetrates down to copper or copper alloys. Since nickel is not attacked by moist sulfur vapor at 100°C or less, this test will not detect pores or

cracks in the top coating if such pores or cracks do not penetrate through the nickel underplate overlaying the copper.

5.6 The level of porosity in the coating that may be tolerable depends on the severity of the environment that the product is likely to encounter during service or storage. Also, the location of the pores on the surface is important. If the pores are few in number or away from the significant surfaces, their presence can often be tolerated.

5.7 The present test method can be used on samples of various geometries, such as curved surfaces. It can also be used for selective area coatings, if allowance is made for tarnish creepage from bare copper alloy areas.

5.8 This test method is destructive in that it reveals the presence of porosity by contaminating the surface with tarnish films. Any parts exposed to this test method should not be placed in service.

5.9 The relationship of porosity levels revealed by this test method to product performance and service life must be made by the user of the test through practical experience or by judgment. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores on the significant surfaces may be acceptable for others.

6. Apparatus

6.1 *Test Vessel*—May be any convenient-size vessel of glass, acrylic-resin (or of any other material that is not affected by high humidity or sulfur), such as a glass desiccator of 9 to 10 L capacity. It should have a lid or cover capable of being plugged with a stopper. The stopper shall have a 1 to 4 mm diameter hole through it to serve as a vent.

6.2 *Sample Fixture or Holders*—Supports or hangers shall be made from material such as glass or acrylic plastic that will not be affected by sulfur or high humidity, and shall be arranged so that the samples will be at least 75 mm away from the humidity controlling solution or sulfur powder (see 6.3). The samples shall also be at least 25 mm from the vessel walls and at least 10 mm from other samples or other surfaces. Do not use a desiccator plate. The fixture shall not cover more than 20 % of the vessel's cross-sectional area so that air movement within the vessel will not be restricted during the test.

6.3 *Glass Dish*—Petri or other shallow dish approximately 15 cm in diameter to hold powdered sulfur. Dish may be supported above the constant humidity solution with plastic blocks, or floated on the liquid.

6.4 *Oven, Air-circulating*, capable of maintaining test vessel at a temperature of $50 \pm 2^\circ\text{C}$ ($122 \pm 4^\circ\text{F}$).

6.5 *Temperature and Relative Humidity (RH) Sensor*, with a remote sensor probe having a range of approximately 76 to 95 % RH at 50°C, which can be kept in the desiccator during test.⁶

6.6 *Microscope, Optical, Stereo, 10 ×*—It is preferred that one eyepiece contain a graduated reticle for measuring the diameter of tarnish spots. The reticle shall be calibrated for the magnification at which the microscope is to be used.

6.7 *Light Source*, incandescent or circular fluorescent.

⁶ The Hydrodynamics Hygrometer, manufactured by Newport Scientific, Inc., has been found satisfactory for this purpose.