



# Standard Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor<sup>1</sup>

This standard is issued under the fixed designation B 799; 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 equipment and methods for determining the porosity of gold and palladium coatings, particularly electrodeposits and clad metals used on electrical contacts.

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

1.3 A variety of other porosity testing methods are described in the literature.<sup>2,3</sup> Other porosity test methods are B 735, B 741, B 798, and B 809. An ASTM Guide to the selection of porosity tests for electrodeposits and related metallic coatings is available as Guide B 765.

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

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 establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazards, see Section 6.

## 2. Referenced Documents

### 2.1 ASTM Standards:

B 374 Terminology Relating to Electroplating<sup>4</sup>

B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor<sup>5</sup>

B 741 Test Method for Porosity In Gold Coatings on Metal

Substrates by Paper Electrography<sup>5</sup>

B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings<sup>4</sup>

B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography<sup>5</sup>

B 809 Test Method for Porosity in Metallic Coatings By Humid Sulfur Vapor (“Flowers-of-Sulfur”)<sup>4</sup>

## 3. Terminology

3.1 Definitions—Many terms used in this test method are defined in Terminology B 542 and terms relating to metallic coatings are defined in Terminology B 374.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *corrosion products*—those reaction products emanating from the pores that protrude from, or are otherwise attached to, the coating surface after a vapor test exposure.

3.2.2 *measurement area (or “significant surface”)*—the surface that is examined for the presence of porosity. The significant surfaces or measurement areas of the part to be tested shall be indicated on the drawing of the part or by provision of suitably marked samples.

3.2.3 *Discussion*—For specification purposes, the significant surfaces or measurement areas are often defined as those portions of the surface that are essential to the serviceability or function of the part, such as its contact properties, or which can be the source of corrosion products or tarnish films that interfere with the function of the part.

3.2.4 *metallic coatings*—include platings, claddings, or other metallic layers applied to the substrate. The coatings can comprise a single metallic layer or a combination of metallic layers.

3.2.5 *Porosity*—the presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal.

3.2.6 *Underplate*—a metallic coating layer between the substrate and the topmost layer or layers. The thickness of an underplate is usually greater than 0.8 μm (30 μin.).

## 4. Summary of Test Method

4.1 The test method employs concentrated sulfurous acid (H<sub>2</sub>SO<sub>3</sub>), which emits sulfur dioxide (SO<sub>2</sub>) gas according to the equilibrium reaction:

<sup>1</sup> 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.

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<sup>2</sup> For example see: Nobel, F. J., Ostrow, B. D., and Thompson, D. W., “Porosity Testing of Gold Deposits,” *Plating*, Vol 52, 1965, p. 1001.

<sup>3</sup> S. J. Krumbien, Porosity Testing of Contact Platings, *Proceedings, Connectors and Interconnection Technology Symposium*, Oct. 1987, p 47.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 02.05.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 02.04.



The procedure is similar to one first proposed by Lee and Ternowski.<sup>6</sup>

4.2 Exposure periods may vary, depending upon the degree of porosity to be revealed. Reaction of the gas with a corrodable base metal at pore sites produces reaction products that appear as discrete spots on the gold or palladium surface. Individual spots are counted with the aid of a loupe or low-power stereo microscope.

4.3 This test method is suitable for coatings containing 95 % or more of gold or palladium on substrates of copper, nickel, and their alloys which are commonly used in electrical contacts.

4.4 This porosity test involves 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 the electrical performance of contacts unless correlation is first established with service experience.

## 5. Significance and Use

5.1 Gold coatings are often specified for the contacts of separable electrical connectors and other devices. Electrodeposits are the form of gold that is most used on contacts, although it is also employed as inlay or clad metal and as weldments on the contact surface. The intrinsic nobility of gold enables it to resist the formation of insulating oxide films that could interfere with reliable contact operation.

5.2 Palladium coatings are sometimes specified as alternatives to gold on electrical contacts and similar electrical component surfaces, both as electrodeposits and as inlay or clad metal. This test method is particularly suitable for determining porosity in palladium coatings, since the reactive atmosphere that is used does not attack the palladium if the specified test conditions are followed. In contrast, palladium coatings are attacked by nitric acid (HNO<sub>3</sub>) and other strong oxidizing agents, so that Test Method B 735 cannot be used for determining the porosity in such coatings.

5.3 In order for these coatings to function as intended, porosity, cracks, and other defects in the coating that expose base-metal substrates and underplates must be minimal or absent, except in those cases where it is feasible to use the contacts in structures that shield the surface from the environment or where corrosion inhibiting surface treatments for the deposit are employed. The level of porosity in the coating that may be tolerable depends on the severity of the environment to the underplate or substrate, design factors for the contact device like the force with which it is mated, circuit parameters, and the reliability of contact operation that it is necessary to maintain. Also, when present, the location of pores on the surface is important. If the pores are few in number and are outside of the zone of contact of the mating surfaces, their presence can often be tolerated.

5.4 Methods for determining pores on a contact surface are most suitable if they enable their precise location and numbers to be determined. Contact surfaces are often curved or irregular in shape, and testing methods should be suitable for them. In addition, the severity of porosity-determining tests may vary from procedures capable of detecting all porosity to procedures that detect only highly porous conditions.

5.5 The present test method is capable of detecting virtually all porosity or other defects that could participate in corrosion reactions with the substrate or underplate. The test is rapid, simple, and inexpensive. In addition, it can be used on contacts having complex geometry such as pin-socket contacts (although with deep recesses it is preferred that the contact structures be opened to permit reaction of the sulfur dioxide with the interior significant surfaces).

5.6 The relationship of porosity levels revealed by particular tests to contact behavior must be made by the user of these tests through practical experience or by judgment. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores in the contact zone may be acceptable for others.

5.7 This test is considered destructive in that it reveals the presence of porosity by contaminating the surface with corrosion products and by undercutting the coating at pore sites or at the boundaries of the unplated areas. Any parts exposed to this test shall not be placed in service.

5.8 This test is intended to be used for quantitative descriptions of porosity (such as number of pores per unit area or per contact) only on coatings that have a pore density sufficiently low that the corrosion sites are well separated and can be readily resolved. As a general guideline this can be achieved for pore densities up to about 100/cm<sup>2</sup>. Above this value the tests are useful for the qualitative detection and comparisons of porosity.

5.9 For these purposes, the *measurement area*, or *significant surface*, shall be defined as those portions of the surface that are essential to the serviceability or function of the part, such as its contact properties, or which can be the source of corrosion products or tarnish films that interfere with the function of the part. The significant surfaces shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

## 6. Safety Hazards

6.1 Carry out these test procedures in a clean, working fume hood. The SO<sub>2</sub> gas that is emitted is toxic, corrosive, and irritating.

6.2 Use caution, however, in actually performing the tests that the drafts often found in hoods do not cause significant cooling of the chamber walls which may lead to condensation of water and acceleration of the test. It is often convenient to enclose the reaction vessel in a box with a loose-fitting cover, and to keep the box in a hood during the test.

6.3 Observe normal precautions in handling corrosive acids. In particular, wear eye protection completely enclosing the eyes, and make eye wash facilities readily available.

<sup>6</sup> Lee, F., and Ternowski, M., *Proceedings Ninth International Conference on Electrical Contact Phenomena*, Chicago, 1978, p. 215.

## 7. Apparatus

7.1 *Test Chamber*—May be any convenient size, glass or acrylic resin vessel having a gastight lid, such as a glass desiccator of 9 to 10-L capacity. The ratio of chamber volume in cubic centimeters to the generating solution (sulfurous acid) surface area in square centimeters shall not exceed  $25 \pm 1$ .

7.2 *Specimen Holders or Supports*—May be made of glass, polytetrafluoroethylene, or other inert materials. It is essential that the specimens be arranged so as not to impede circulation of the gas. Specimens shall not be closer than 25 mm (1 in.) from the wall and 75 mm (3 in.) from the solution surface. Also, the measurement areas of the specimens shall be at least 12 mm (0.5 in.) from each other.

7.2.1 Do not use a porcelain plate or any other structure that would cover more than 30 % of the liquid surface cross-sectional area. This is to insure that movement of air and vapor within the vessel will not be restricted during the test.

7.3 Stereomicroscope having a 10 $\times$  magnification shall be used for pore counting. In addition a movable source of illumination capable of giving oblique lighting on the specimen surface is required. It is preferred that a graduated reticle be fitted into one of the eyepieces of the microscope.

## 8. Reagents

8.1 *Sulfurous Acid* ( $\text{H}_2\text{SO}_3$ ), “analyzed reagent grade”, or better, preferably in individual sealed 500-mL bottles.

## 9. Procedure

9.1 Handle specimens as little as possible, and only with tweezers, microscope-lens tissue, or clean, soft cotton gloves. Prior to the test, inspect the samples under 10 $\times$  magnification for evidence of particulate matter. If present, such particles shall be removed by blowing them with clean, oil-free air. If this is not successful discard the sample. Then, clean the samples with solvents or solutions that do not contain chlorinated hydrocarbons, CFC's or other known ozone-destroying compounds. The procedure outlined in Note 1 has been found to give satisfactory results for platings with mild to moderate surface contamination.

NOTE 1—Suggested cleaning procedure:

- (1) Keep individual contacts separated if there is a possibility of damage to the measurement areas during the various cleaning steps.
- (2) Clean samples for 5 min in an ultrasonic cleaner which contains a hot (65–85°C) 2 % aqueous solution of a mildly alkaline (pH 7.5–10) detergent (such as Micro or Sparkleen).
- (3) After ultrasonic cleaning, rinse samples under warm running tap water for at least 5 s.
- (4) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.
- (5) Immerse in fresh methanol or isopropanol, and ultrasonically “agitate” for at least 30 s in order to remove the water from the samples.
- (6) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid to drying, the air shall be oil free, clean, and dry.
- (7) Do not touch measurement area of the samples with bare fingers after cleaning.
- (8) Re-inspect samples (under 10 $\times$  magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning steps. Surface cleanliness is extremely important; contaminants, such as plating salts, organic films, and metal flakes may give erroneous indications of defects, and are unacceptable.

NOTE 2—Omit the cleaning steps for samples having corrosion-inhibiting, or lubricant coatings, or both, if it is desired to determine the efficacy of these coatings in the  $\text{SO}_2$  atmosphere.

9.2 The test temperature shall be  $23 \pm 3^\circ\text{C}$ , unless otherwise specified, and the relative humidity in the immediate vicinity of the test chamber shall be no greater than 60 %. If the relative humidity is greater than 60 %, do not run the test.

9.3 Carefully add the sulfurous acid ( $\text{H}_2\text{SO}_3$ ) to the bottom of the *clean* and *dry* test chamber *in a fume hood*, load the samples, using suitable fixtures, and replace the lid. The ambient relative humidity shall be no greater than 60 % during both the addition of the  $\text{H}_2\text{SO}_3$  and the insertion of the samples.

9.4 Expose the samples to the test environment for 2 h for gold coating thicknesses of 1.25  $\mu\text{m}$  (50  $\mu\text{in.}$ ) or greater, 90 min for gold thicknesses less than 1.25  $\mu\text{m}$ , and 60 min for palladium coatings.

9.5 Remove the samples at the end of the test period, and allow them to stabilize at low-humidity under the fume hood for  $10 \pm 5$  min.

9.6 Place samples (preferably with their holding fixture, if possible) into an air circulating oven, maintained at  $125 \pm 5^\circ\text{C}$ , for  $15 \pm 5$  min, for the purpose of “developing” the pore-corrosion products for easier examination.

9.7 Discard the sulfurous acid in a safe manner at the end of each day. It may be re-used during any one day for several consecutive tests provided the chamber is kept closed between test runs, and no discoloration or contamination, visible to the naked eye, is present.

## 10. Examination

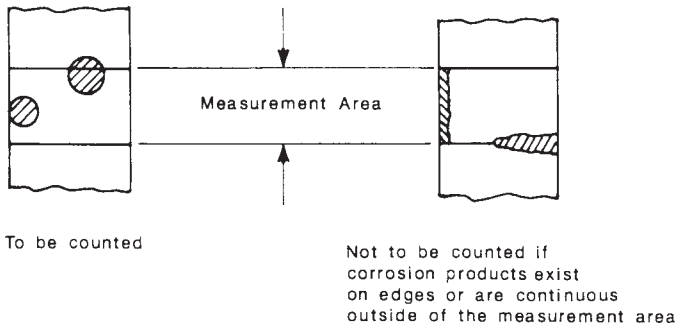
10.1 Count individual pore-corrosion products at 10 $\times$  magnification, using collimated incandescent illumination at an oblique angle below  $15^\circ$ . They are delineated by the corrosion products protruding from the pore sites. The solids may be transparent, especially in the case of gold or palladium-plated nickel; exercise great care in counting, particularly for rough or curved surfaces.

NOTE 3—The following hints may be useful as an aid to counting.

- (1) Count only corrosion products that protrude above the surface. Stains are not considered porosity within the scope of this specification.
- (2) Loose contamination that can easily be removed by a gentle air dusting should not be considered corrosion products.
- (3) Move sample around under the light to vary the angle when unsure of a pore. Burnished gold can give the appearance of black spots.
- (4) A corrosion product should be measured and counted when at least one half of the corrosion product falls within the *measurement area*. Unless otherwise specified, corrosion products which initiate outside the measurement area but fall within it, and which are irregular in shape should not be counted, see Fig. 1. However, for small measurement areas, or where the migrating pore-corrosion product covers a significant portion of this area, the presence of such products should be recorded.

10.2 Pore size shall be defined by the longest diameter of the corrosion product. Unless otherwise specified, corrosion products less than 0.05 mm (0.002 in.) in diameter shall not be counted. A graduated reticle in the microscope eyepiece is useful as an aid to counting and sizing.

NOTE 4—A useful sizing technique is to tabulate the pore-corrosion products in accordance with three size ranges. These are (approximately): (a) 0.12 mm diameter (0.005 in.) or less, (b) between 0.12 and 0.40 mm



**FIG. 1 Corrosion Products at Boundaries of Measurement Area**

diameter (0.005 and 0.015 in.), and (c) greater than 0.40 mm diameter (0.015 in.).

10.3 The acceptable number, sizes and locations of the pore corrosion products shall be as specified on the appropriate drawing or specification, or as agreed upon by the producer and user.

## 11. Precision and Bias

11.1 *Precision*—The precision of this test method is being investigated with gold-plated electrical contacts having a

nickel underplate. Round-robin results from a series of four runs in each laboratory with an SO<sub>2</sub> test of greater severity gave coefficients of variation of less than 20 % in each of three participating laboratories. However, poorer precision was obtained when the results of the different laboratories were compared.

11.2 *Bias*—The porosity of commercially produced contact platings is a property with potentially large sample-to-sample variability.<sup>7</sup> Since there is no acceptable reference material suitable for determining the bias for porosity testing, no statement on bias is being made.

## 12. Keywords

12.1 gold coatings; gold platings; palladium coatings; palladium platings; plating porosity; porosity testing; pore corrosion test; sulfur dioxide test; sulfurous acid/SO<sub>2</sub> test

<sup>7</sup> Krumbein, S. J., and Holden, C. A., Jr., "Porosity Testing of Metallic Coatings," in "Testing of Metallic and Inorganic Coatings," *ASTM STP No. 947*, Harding, W. B., and DiBari, G. A., Eds., ASTM, PA, 1987, p. 193.

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