



# Standard Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography<sup>1</sup>

This standard is issued under the fixed designation B798; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers equipment and techniques for determining porosity in noble metal coatings, particularly electrodeposits and clad metals used on electrical contacts.

1.2 The 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 Other porosity testing methods are outlined in Guide B765. Detailed critical reviews of porosity testing are also available.<sup>2</sup> Other porosity test methods are B735, B741, B799, and B809.

1.4 The values stated in SI units are to be regarded as standard. The values 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 become familiar with all hazards including those identified in the appropriate Material Safety Data Sheet (MSDS) 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.* For specific hazard statements, see Sections 7 and 8.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

B374 Terminology Relating to Electroplating

B542 Terminology Relating to Electrical Contacts and Their Use

<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> Nobel, F. J., Ostrow, B. D., and Thompson, D. W., "Porosity Testing of Gold Deposits," *Plating*, Vol 52, 1965, p. 1001, and Krumbain S. J., "Porosity Testing of Contact Platings," *Proceedings, Connectors and Interconnection Technology Symposium*, October 1987, p. 47.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

B735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor

B741 Test Method for Porosity In Gold Coatings On Metal Substrates By Paper Electrography (Withdrawn 2005)<sup>4</sup>

B765 Guide for Selection of Porosity and Gross Defect Tests for Electrodeposits and Related Metallic Coatings

B799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor

B809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers-of-Sulfur")

## 3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminology B542 and terms relating to metallic coatings are defined in Terminology B374.

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

3.2.1 *decorations, n*—those reaction products emanating from the pores that provide visual contrast with the gel medium.

3.2.2 *measurement area (or "significant surface"), n*—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.

3.2.2.1 *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.

3.2.3 *metallic coatings, n*—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.

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

3.2.5 *underplate, n*—a metallic coating layer between the substrate and the topmost layer or layers. The thickness of an underplate is usually greater than 0.8  $\mu\text{m}$  (30  $\mu\text{in.}$ ).

<sup>4</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

#### 4. Summary of Test Method

4.1 This test method is an electrographic technique, “gel-bulk electrography.” The specimen is made the anode in a cell containing a solid or semisolid electrolyte of gelatin, conducting salts, and an indicator. Application of current to this cell results in the migration of base metal ions through continuous pores. Reaction of cations with an indicator gives rise to colored reaction products at pore sites which may be counted through the clear gel. Individual spots are counted with the aid of a loupe or low power stereomicroscope.

4.2 This test method is suitable for coatings containing 75 % or more of gold on substrates of silver, nickel, copper, and its alloys, which are commonly used in electrical contacts. This test method is also suitable for coatings of 95 % or more of palladium on nickel, copper and its alloys.

4.3 These porosity tests involve corrosion reactions in which the products delineate defect sites in coatings. Since the chemistry and properties of these products do not resemble those found in natural or service environments, these tests are not recommended for prediction of the electrical performance of contacts unless correlation is first established with service experience.

#### 5. Significance and Use

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

5.2 In order that the nobility of gold be assured, 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 or are outside of the zone of contact of the mating surfaces, their presence can often be tolerated.

5.3 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 gross defects. The test method in this document is generally regarded as severe.

5.4 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 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.

5.5 This test method is capable of detecting porosity or other defects in gold or palladium coatings that could participate in substrate corrosion reactions. In addition, it can be used on contacts having complex geometry such as pin-socket contacts (although difficulty may be experienced in inspecting deep recesses).

#### 6. Limitations

6.1 This test is considered destructive in that it reveals the presence of porosity by contaminating the surface with corrosion products and by under-cutting the corrodible metal at pore sites and at unplated areas. In addition, the surface is coated with a corrosive gel mixture which is difficult to remove completely. Any parts exposed to the gel test shall not be placed in service.

6.2 The gel-bulk procedure is not as sensitive to small pores and is more complex than porosity tests involving gaseous corrodants<sup>5</sup> (see Test Methods **B735** and **B799**). It also involves more chemicals, preparation, and auxiliary equipment.

6.3 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 measurement areas where coatings have pore densities that are sufficiently low so 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 25/cm<sup>2</sup>.

6.4 For this purpose, 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. When necessary, the significant surfaces shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

6.5 The test applicability to platings of varying thickness is a function of the quality of the plating.

6.6 The applicability of this test method to localized platings or claddings with adjacent exposed substrate is limited by the efficacy of coatings applied to mask the non-noble areas to prevent gross decoration of the surfaces under test. Users of this method are required to develop their own techniques for masking such exposed substrate areas.

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<sup>5</sup> For example, Clarke, M., “Porosity and Porosity Tests,” in “Properties of Electrodeposits,” edited by Sard, Leidheiser, and Ogburn, The Electrochemical Society, 1975, p. 122.

## 7. Apparatus

7.1 *Test Vessel* may be made of glass, acrylic resin, or other inert uncolored transparent material. It shall have thin-walled flat sides, and be of a size appropriate to the sample to be tested.

7.2 *Power Supply*, 0 to 1 A and 0 to 10 V dc, an electronically-regulated, constant-current ( $\pm 5\%$ ) apparatus is preferred.

7.3 *dc Milliammeter and Separate dc Voltmeter*.

7.4 *Cathode Material* in the form of foil or wire made of platinum or gold is required. The cathode and specimen (anode) areas shall be approximately the same. Additionally, gold or platinum wire for cathode and anode are needed for that portion of the hook-up that is in the reagent solution. It may be convenient to use small alligator clips to secure the lead wires to the cathode and anode. These clips must be heavily gold plated so as to be entirely free of porosity. A variation of this procedure, suitable for samples having relatively few pores, is to use a second identical test sample as the cathode. The test can be run with current first in the forward, then in the reverse direction so that the porosity in both samples may be determined. Fig. 1 is a schematic of the test cell setup.

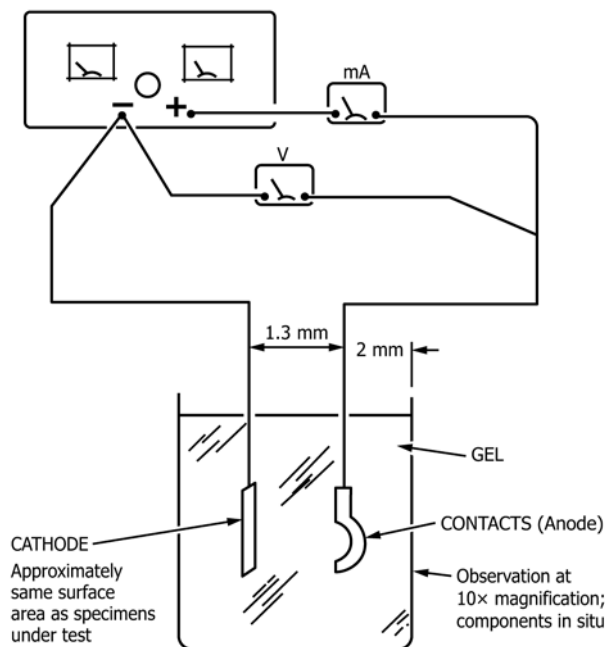


FIG. 1 Schematic of Typical Test-Cell Setup with Anode (Sample) and Cathode Facing Each Other (Preferred Orientation)

NOTE 1—A commonly-used alternate cell design incorporates the cathode as part of the cell structure (as shown in Fig. 2). In addition, the samples may be attached to a common carrier strip or holder, so that only the sample surfaces need be in the gel.

7.5 *Timer* capable of indicating seconds. It is convenient to use a timer switch to control the test current.

7.6 *Stereomicroscope* having 10 $\times$  magnification and an illuminator are required for sample inspection after test. An

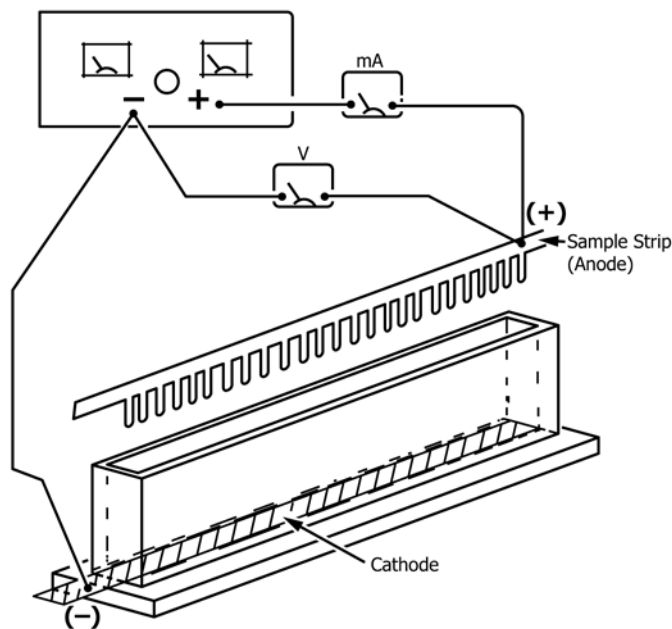


FIG. 2 Exploded View of Alternate Cell Design Incorporating Cathode as Part of Cell Structure

eyepiece reticle is recommended for convenience in locating the contact area or other significant measurement areas.

## 8. Reagent

8.1 Note that some of the indicating reagents are sensitive to heat and light, particularly the rubenic acid (dithio-oxamide). The indicator solutions should be stored in the dark in stoppered bottles. For rubenic acid, do not store for more than a month, and filter prior to use.

8.2 *Food-Grade Gelatin*—This type is preferred to USP grade gelatin, because the latter may not give transparent solutions. A 10% solution is prepared by mixing 9 g of the gelatin in 91 mL of distilled or deionized water, and slowly heating to 60 to 65°C with stirring, until all the gelatin dissolves.

NOTE 2—If the storage bottle is tightly capped, the plain gelatin solution may be stored for up to 2 days in a refrigerator and kept at 5 to 10°C, discard it if mold appears on its surface.

## 9. Safety Hazards

9.1 Reagents identified in Table 1 have the potential to cause injury or skin discoloring if improperly handled. Good laboratory practice including the use of a fume hood and skin and eye protection should be observed, especially during solution preparative and the cleaning of the test samples. Proper precautions in the use of electrical power supplies and electrical connections should also be scrupulously observed.

## 10. Procedure

10.1 This test is suitable for gold coated on silver, nickel, or copper and its alloys, and palladium coated on nickel, copper and its alloys either as underlayers or substrates, in accordance with the reagents chosen in Table 1.

10.2 *Sequence of Operations*:

**TABLE 1 Guide to Gel Porosity Testing Solutions**

Test for	Electrolyte (Aqueous)	Indicator	Indicating Color	Comments
Copper <sup>A</sup>	4 % sodium carbonate + 1 % sodium nitrate	saturated solution of rubeanic acid in ethanol	dark olive green	also detects nickel, cobalt
Copper	4 % sodium carbonate + 1 % sodium nitrate	7.5 % potassium ferrocyanide in water	brown	-----
Nickel <sup>A</sup>	4 % sodium carbonate + 1 % sodium nitrate	saturated solution of rubeanic acid in ethanol	blue-blue violet	also detects copper, cobalt
Nickel	5 % ammonium hydroxide	saturated solution of dimethylglyoxime in ethanol	pink	-----
Silver <sup>A, B</sup>	0.2 molar nitric acid	1 % glacial acetic + 5 % sodium dichromate	red	solution must be free of halogens
Silver <sup>B</sup>	0.2 molar nitric acid	saturated solution of rhodanine in ethanol	red-red violet	solution must be free of halogens

<sup>A</sup> Preferred test.

<sup>B</sup> Not suitable for palladium overplates.

### 10.2.1 Solution Preparation:

#### 10.2.1.1 Electrolyte.

#### 10.2.1.2 Indicator.

10.2.2 Calculate the current to be used.

10.2.3 Prepare the samples prior to cleaning.

10.2.4 Clean the samples.

10.2.5 Prepare the gel while the samples are cleaning.

Remove from heat when dissolved.

10.2.6 Dry the samples.

10.2.7 Suspend the samples in the test cells.

10.2.8 Prepare the composite gel solution and add to the cells.

10.2.9 Solidify the gel thoroughly.

10.2.10 Set up the equipment and make electrical attachments.

10.2.11 Apply the calculated current.

10.2.11.1 Examine immediately for gross defects.

10.2.11.2 Take readings after the prescribed time period.

10.2.12 Record the results.

10.3 *Cleaning*—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× magnification for evidence of particulate matter. If present, such particles shall be removed by blowing them off 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, CFSs or other known ozone-destroying compounds. The procedure outlined in **Note 3** has been found to give satisfactory results for platings with mild to moderate surface contamination.

**NOTE 3**—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× 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.

10.4 Secure the specimens to be tested to gold or platinum lead wires, and suspend in the cell so that their measurement areas (see **5.4**) can be easily seen through the wall of the test vessel. Place the specimens  $13 \pm 2$  mm (0.5 in.) from the cathode.

**NOTE 4**—For longitudinal-shaped contacts, such as pins or posts, where the alternate cell design (**Fig. 2**) is used the bottom ends or tips of the contacts should be at a distance from the cathode that is at least 1.5 times their maximum length in the gel solution. The area of the cathode along the bottom of the cell shall be no less than the total specimen area in the solution, and can be up to twice as large. If the cathode area is too small, there is a risk of excessive hydrogen bubble formation.

10.5 The composite test solution is prepared from the gel solution, and an indicator and electrolyte chosen from **Table 1**. The composite contains by volume, 94.6 % gel solution, 3.4 % electrolyte solution, and 2 % indicating solution. It is prepared by melting with gentle stirring, the gelatin solution at  $59^\circ\text{C} \pm 1^\circ\text{C}$ , removing from the heat, and adding while stirring the premixed electrolyte solution and then the indicating solution. Stirring should be continued for 2 to 3 min to thoroughly mix all the components. For consistency, consider the lifespan of the composite gel solution to be 2 h. Testing of the samples shall be accomplished within this time period.

10.6 Pour the composite test gel solution into the test vessel, taking care not to introduce bubbles of air into the solution. Allow the gel to solidify.

**NOTE 5**—It is good practice to include with the sample a control specimen that is known to be porous (such as would be produced by scratching the coating to expose base metal.) If the control specimen fails to produce a dense opaque pore indication the test results must be discounted and the test repeated using freshly prepared reagents.

10.7 Attach the specimen lead to the positive terminal of the power supply, and the cathode lead to its negative terminal, as shown in **Fig. 1** or **Fig. 2**.

10.8 Pass the current for 20 s (use a timer) at a current density of  $0.78 \text{ ma/cm}^2$  ( $5 \text{ ma/in.}^2$ ). Current density tolerance



is  $\pm 25\%$ . The test shall be conducted at  $23 \pm 3^\circ\text{C}$ . During passage of the current, base metal ions are produced at pores in the test sample. These ions react with the indicator to produce colored products in the pore sites.

NOTE 6—If bubbling of the gel should occur (highly unlikely at the low current density specified), repeat the test with new samples at 20 to 25 % lower current density.

NOTE 7—In calculating the current, the sample area shall be the *total* area of metal that is immersed in the gel, not just the significant surfaces.

10.9 Monitor and record the maximum voltage with the voltmeter during the application of current. The voltage should be between 2.0 and 2.5 V. If the voltage exceeds 3 V, the test should be considered invalid.

## 11. Examination

11.1 Do not remove samples from the test vessel. Examine the measurement areas of the samples through the vessel walls (at 10 $\times$  magnification with suitable illumination) immediately after cessation of current. Note the gross defects during the first 3 min to determine the general location of the largest pore sites.

11.2 Begin actual pore count after 3 min, and complete it within 20 min of cessation of the current.

11.3 Pore sites show up as colored dots or blooms. The size of these decorations depends on the total amount of current passed as well as the size of the defect. Bleeding or diffusion of the color should be slight during the first 20 min, and discrete pore sites will be observed if not very close to each other. Cracks show up as continuous lines.

NOTE 8—Appendix X1 gives a number of guidelines that have been found useful in examining pore indications from tests that use the rubeanic acid indicator and the sodium carbonate/sodium nitrate electrolyte (Table 1).

11.4 An alternative enumeration method is to record the number of contacts with pores (in a sample strip with 10 or more contacts), as defined in 11.3

## 12. Precision and Bias

12.1 *Precision*—The precision of this procedure for five test runs in one laboratory, for palladium platings 2.2–2.4  $\mu\text{m}$  thick, was 46 % (as a coefficient of variation where the total number of pores per run had a mean value of 2.8) and was even higher in subsequent determinations in other laboratories.

12.2 *Bias*—The porosity of commercially produced platings for electrical contacts is a property with potentially large sample-to-sample variability.<sup>6</sup> Since there is no acceptable reference material suitable for determining the bias for porosity testing, bias has not been determined.

## 13. Keywords

13.1 electrography; gel; gel electrography; gold coatings; gold platings; palladium coatings; palladium platings; plating; porosity; porosity testing

<sup>6</sup> Krumbein, S. J., and Holden, C. A., Jr., "Porosity Testing of Metallic Coatings," in "Testing of Metallic and Inorganic Coatings," *ASTM, STP 947*, Harding, W. B., and DiBari, G. A., Eds., American Society for Testing and Materials, PA, 1987, p. 193.

# APPENDIX

## (Nonmandatory Information)

### X1. USEFUL GUIDELINES FOR EXAMINING EXAMPLES

X1.1 The following guidelines are useful in examining samples (for pore indications) with tests that use the rubeanic acid indicator and sodium carbonate/sodium nitrate electrolyte (of Table 1).

X1.1.1 The reaction products give colored blooms whose major growth takes place during the first 3 min after cessation of current. Blooms from the edges of narrow samples (for example, posts) may obscure the measurement area, so the immediate cursory examination is important to determine pore sites. A typical site usually shows a dark spot at the center of a colored cloud.

X1.1.2 The indicator will react with nickel and copper ions, producing colored blooms. Copper will often show an olive or gray-green reaction product while nickel will often produce a blue or blue-violet coloration.

X1.1.3 When a nickel underplate is used over a copper alloy substrate, two colors may be present. Nickel ions diffuse farther (away from the pore) and may form a blue to violet haze around the central olive-green or black central copper pore indication.


X1.1.4 Under certain conditions, palladium platings may produce bright red/orange reaction products. These blooms shall not be counted as pores, unless evidence of nickel or copper pore indications are also present.

X1.1.5 Very small pores and embedded debris show up as small colored points or dots at 10 $\times$  and, shall be recorded separately as such.

X1.1.6 Cracks show up as continuous lines and shall be recorded separately as such.

X1.1.7 A hazy black cloud covering the surface without evidence of dark spots (pore sites) indicates gross defects or microcracking, and shall be counted as a multiple pore site.

X1.1.8 When plated electrical contacts are tested together as strips or combs of contacts, the size of a pore indication is most conveniently noted as a fraction of the width of the contact surface.

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