Designation: B798 – 95 (Reapproved 2014)

Standard Test Method for
Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography[1]

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 (ε) 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.[2] 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:*[3]
B374 Terminology Relating to Electroplating
B542 Terminology Relating to Electrical Contacts and Their Use

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B741 Test Method for Porosity In Gold Coatings On Metal Substrates By Paper Electrography (Withdrawn 2005)[4]
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 that 0.8 µm (30 µin.).

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[2] Nobel, F. J., Ostrow, B. D., and Thompson, D. W., "Porosity Testing of Gold Deposity," *Plating*, Vol 52, 1965, p. 1001, and Krumbein S. J., "Porosity Testing of Contact Platings," *Proceedings, Connectors and Interconnection Technology Symposium*, October 1987, p. 47.

[3] 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.

[4] The last approved version of this historical standard is referenced on 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 medal 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[5] (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 $25/cm^2$.

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.

[5] 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 (±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.
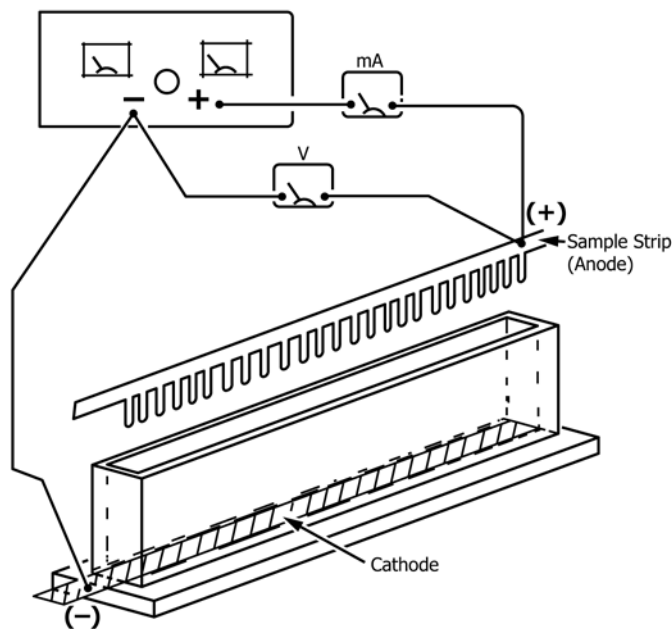

FIG. 2 Exploded View of Alternate Cell Design Incorporating Cathode as Part of Cell Structure

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× magnification and an illuminator are required for sample inspection after test. An 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 rubeanic acid (dithio-oxamide). The indicator solutions should be stored in the dark in stoppered bottles. For rubeanic 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.
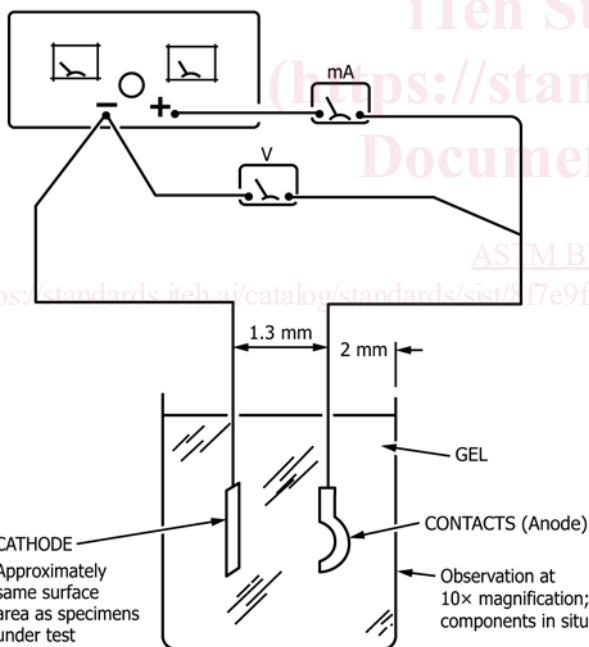
10.2 *Sequence of Operations:*