Designation: B920 − 16 (Reapproved 2022)

Standard Practice for
Porosity in Gold and Palladium Alloy Coatings on Metal Substrates by Vapors of Sodium Hypochlorite Solution[1]

This standard is issued under the fixed designation B920; 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 practice covers equipment and methods for revealing the porosity of gold and palladium coatings, particularly electrodeposits and clad metals used on electrical contacts.

1.2 This test practice is suitable for coatings containing gold or 75 % by mass of palladium on substrates of copper, nickel, and their alloys, which are commonly used in electrical contacts.

1.3 A variety of full porosity testing methods is described in the literature.[2,3] These porosity Test Methods are B735, B741, B798, B799, and B809. An ASTM Guide to the selection of porosity tests for electrodeposits and related metallic coatings is available as Guide B765.

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

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:*[4]
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B741 Test Method for Porosity In Gold Coatings On Metal Substrates By Paper Electrography (Withdrawn 2005)[5]
B765 Guide for Selection of Porosity and Gross Defect Tests for Electrodeposits and Related Metallic Coatings
B798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography
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 practice 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 *corrosion products, n*—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 *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.

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

3.2.4 *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 µm (30 µin.).

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[1] This practice is under the jurisdiction of ASTM Committee B02 on Nonferrous Metals and Alloys and is the direct responsibility of Subcommittee B02.05 on Precious Metals and Electrical Contact Materials and Test Methods.
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[2] For example see: Nobel, F. J., Ostrow, B. D., and Thompson, D. W., "Porosity Testing of Gold Deposits," *Plating*, Vol 52, 1965, p. 1001.
[3] Krumbien, S J., Porosity Testing of Contact Platings, Proceedings, Connectors and Interconnection Technology Symposium, Oct. 1987, p. 47.

[4] 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] The last approved version of this historical standard is referenced on www.astm.org.

4. Summary of Practice

4.1 The test practice employs a solution of sodium hypochlorite, a material readily available as household bleach. The test is recommended primarily as a qualitative means for assessing the plating quality in electrical connectors and is desirable because it uses readily available reagents and equipment and is extremely inexpensive, simple, and fast. In the test, the coated parts to be evaluated are suspended above a solution of sodium hypochorite in a vessel that is closed but not sealed. Paper towels extend from the solution in the bottom part of the way up the sides of vessel providing a wicking action for the solution. After exposure in this manner for 30 min to 45 min, the parts are removed from the vessel, dried with hot air and examined for the presence of corrosion products that indicate porosity.

4.2 For more quantitative characterization of porosity it is better to use one of the previously mentioned porosity test standards. This practice is oftentimes used as an early predictor of the likelihood of failure in a full mixed flowing gas (MFG) test used as an accelerated environmental test.

4.3 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 may be counted with the aid of a loupe or low-power stereomicroscope.

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 Palladium and 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 and palladium alloys enables it to resist the formation of insulating oxide films that could interfere with reliable contact operation.

5.2 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.

5.3 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 highly porous conditions.

5.4 The present test practice is capable of detecting virtually all porosity or other defects that could participate in corrosion reactions with the substrate or underplate. 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 vapors with the interior significant surfaces).

5.5 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 other forms of testing. 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. The acceptable number, sizes and locations of the pore corrosion products shall be as specified on the appropriate drawing or specification.

5.6 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.

5.7 The test is simple and inexpensive. The cost associated with the test is very low, using standard basic equipment found in an industrial laboratory. There are minimal waste disposal issues associated with the procedure. The test is very popular because of its very quick means of assessing the likelihood of plating quality problems, prior to the performance of accelerated environmental testing on the 1 to 2 week scale at much greater expense.

6. Safety Hazards

6.1 Carry out this test procedure in a clean, working fume hood. The vapor emitted is toxic, corrosive, and irritating.

6.2 Because the test is conducted in a reaction vessel using a loose-fitting cover, it is desirable to insure that the drafts often found in hoods are not so high as to adversely effect the reproducibility of the test within the reaction vessel.

6.3 Observe good laboratory practices when handling the sodium hypochlorite (household bleach) solution. In particular, wear eye protection completely enclosing the eyes, and make eye wash facilities readily available.

7. Apparatus

7.1 *Test Glassware,* a vessel of sufficient size such that the sodium hypochlorite solution at least 1 cm in depth can be placed in the bottom of the vessel without interfering with the samples. The vessel (see Figs. 1 and 2) shall be made of glass
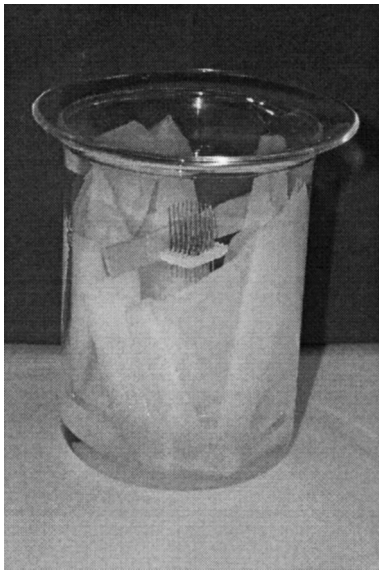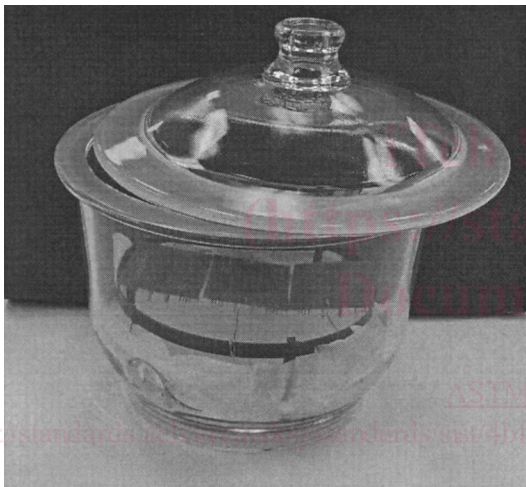
FIG. 1 Test Apparatus Using 400 mL Beaker

FIG. 2 Test Apparatus Using Large Dessicator

or plastic not having a gastight lid, such as a glass beaker with a watch glass cover or desiccator.

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 12 mm (0.5 in.) from the wall or paper towels and 25 mm (1 in.) from the solution surface. Also, the measurement areas of the specimens shall be at least 12 mm (0.5 in.) from each other.

7.3 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.

7.4 *Stereomicroscope,* having at least 20× magnification shall be used for pore examination is recommended. In addition, a movable source of illumination capable of providing oblique lighting on the specimen surface is required. It is

further recommended that a graduated reticle be inserted into one of the eyepieces of the microscope.

7.5 *Hot Air Dryer.*

8. Reagents

8.1 *Sodium Hypochlorite Solution,* 5.25 % nominal (without additives). Acquire this material locally to ensure freshness.

8.2 *Plain Paper Towels,* or suitable non-reactive paper product which will retain its shape following exposure.

9. Procedure

9.1 Handle specimens as little as possible, using only tweezers, microscope-lens tissue, or clean, soft cotton gloves. Prior to the test, inspect the samples under 20× 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*) Dip in methanol if desired to aid in the removal of organic residues; (*3*) Clean samples for 5 min in an ultrasonic cleaner, which contains a hot (65 °C to 85 °C) 2 % aqueous solution of a mildly alkaline (pH 7.5 to 10) detergent (such as Micro or Sparkleen); (*4*) After ultrasonic cleaning, rinse samples under warm running tap water for at least 5 s; (*5*) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues; (*6*) Immerse in fresh methanol or isopropanol, and ultrasonically "agitate" for at least 30 s in order to remove the water from the samples; (*7*) 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; (*8*) Do not touch surface measurement area of the samples with bare fingers after cleaning; (*9*) Re-inspect samples (under 20× 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 filings or 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 test atmosphere.

9.2 The test temperature shall be 23 °C ± 3 °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 %, results can be unpredictable and therefore the test is not acceptable.

NOTE 3—*Procedure to be Performed in Hood or Well-Ventilated Area:* (*1*) Obtain a clean, dry beaker or other vessel (sufficient in size to accommodate your sample size), a cover glass and a specimen support strip, made of an inert material, for example, polyethylene or glass; (*2*) Attach the contacts to be tested to the support strip, by any suitable means such as the use of two-sided adhesive tape so that the areas of interest on the contact extend beyond the edge of the support, and the fumes freely access the surfaces; (*3*) Place strips of paper towel on the inside surface of the vessel, so as to provide a wick for the bleach, and sufficient material for reaction with the solution. (This is important, since the bleach alone will not generate sufficient chlorine to cause a reaction in a short time-frame.); (*4*) Pour in sufficient bleach such that a depth of approximately 1 cm is obtained after the paper towel has been made completely wet. Recap the bottle tightly after use; (*5*) Suspend the contacts 2 cm to 3 cm above the surface of the bleach, and place the cover on the vessel;