



Designation: B 765 – 93 (Reapproved 2001)

## Standard Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings<sup>1</sup>

This standard is issued under the fixed designation B 765; 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 guide describes some of the available standard methods for the detection, identification, and measurement of porosity in electrodeposited and related metallic coatings and provides some laboratory-type evaluations and acceptances. Some applications of the test methods are tabulated in Table 1 and Table 2.

1.2 This guide does not apply to coatings that are produced by thermal spraying, ion bombardment, sputtering, and other similar techniques where the coatings are applied in the form of discrete particles impacting on the substrate.

1.3 This guide does not apply to beneficial or controlled porosity, such as that present in microdiscontinuous chromium coatings.

1.4 Porosity test results occur as chemical reaction end products. Some occur in situ, others on paper, or in a gel coating. Observations are made that are consistent with the test method, the items being tested, and the requirements of the purchaser. These may be visual inspection (unaided eye) or by 10 $\times$  magnification (microscope). Other methods may involve enlarged photographs or photomicrographs.

1.5 The test methods are only summarized. The individual standards must be referred to for the instructions on how to perform the tests.

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

1.7 *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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

B 246 Specification for Tinned Hard-Drawn and Medium-

Hard-Drawn Copper Wire for Electrical Purposes<sup>2</sup>  
B 276 Test Method for Apparent Porosity in Cemented Carbides<sup>3</sup>  
B 374 Terminology Relating to Electroplating<sup>3</sup>  
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure<sup>3</sup>  
B 542 Terminology Relating to Electrical Contacts and Their Use<sup>4</sup>  
B 545 Specification for Electrodeposited Coatings of Tin<sup>3</sup>  
B 605 Specification for Electrodeposited Coatings of Tin-Nickel Alloy<sup>3</sup>  
B 650 Specification for Electrodeposited Engineering Chromium Coatings of Ferrous Substrates<sup>3</sup>  
B 689 Specification for Electroplated Engineering Nickel Coatings<sup>3</sup>  
B 733 Specification for Autocatalytic (Electroless) Nickel-Phosphorous Coatings on Metals<sup>3</sup>  
B 734 Specification for Electrodeposited Copper for Engineering Uses<sup>3</sup>  
B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor<sup>4</sup>  
B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography<sup>4</sup>  
B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography<sup>4</sup>  
B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor<sup>4</sup>  
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers-of-Sulfur”)<sup>3</sup>

### 3. Terminology

3.1 *Definitions*—Many terms used in this guide are defined in Terminology B 374 or B 542.

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

3.2.1 *porosity*—for the purpose of this guide, porosity in a coating is defined as any hole, crack, or other defect that exposes the underlying metal to the environment.

3.2.2 *underplate*—a metallic coating layer between the basis metal and the topmost metallic coating. The thickness of

<sup>1</sup> This guide 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.

Current edition approved Jan. 15, 1993. Published March 1993. Originally published as B 765 – 86. Last previous edition B 765 – 86.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 02.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 02.05.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 02.04.



**TABLE 1 Applications of Standard Porosity Tests to Metallic Coatings (Section 6)**

Substrate Metal <sup>A</sup>	Gold	Silver	Nickel	Tin-Nickel	Tin	Tin-Lead	Copper	Palladium	Chromium
Copper and Copper Alloys	6.1 <sup>B</sup> , 6.2, 6.4, 6.5	6.3A	6.4	6.4	6.4	6.4	...	6.2, 6.3A, 6.4, 6.5	...
Nickel	6.1 <sup>B</sup> , 6.2, 6.5	6.3A	...	...	...	...	...	6.2, 6.3A, 6.5	...
Iron or Steel	6.6	...	6.6	6.3B, 6.6	6.3B, 6.6	6.3B, 6.6	6.6	...	6.6
Silver	6.4	...	6.4	6.4	6.4	6.4	...	6.4	...

<sup>A</sup> The substrate may be the basis metal, an underplate, or both (see Note 1).

<sup>B</sup> Thickness restrictions may apply.

**TABLE 2 Applications of Tests for Gross Defects and Mechanical Damage (Section 7)**

Substrate Metal <sup>A</sup>	Gold	Nickel	Tin-Nickel	Tin	Tin-Lead	Palladium	Silver
Copper and Copper Alloys	7.3, 7.5	7.3, 7.4	7.3	7.3	7.3	7.3, 7.5	7.5
Nickel	7.5	...	...	...	...	7.5	7.5
Iron or Steel	7.1	7.1	7.1	7.1	7.1	7.1	...
Aluminum	...	7.2	...	...	...	...	...

<sup>A</sup> The substrate may be the basis metal, an underplate, or both (see Note 1).

an underplating is usually greater than 1 μm, in contrast to a strike or flash, which are usually thinner.

#### 4. Significance and Use

4.1 Porosity tests indicate the completeness of protection or coverage offered by the coating. When a given coating is known to be protective when properly deposited, the porosity serves as a measure of the control of the process. The effects of substrate finish and preparation, plating bath, coating process, and handling, may all affect the degree of imperfection that is measured.

NOTE 1—The substrate exposed by the pores may be the basis metal, an underplate, or both.

4.2 The tests in this guide involve corrosion reactions in which the products delineate pores in coatings. Since the chemistry and properties of these products may not resemble those found in service environments, these tests are not recommended for prediction of product performance unless correlation is first established with service experience.

#### 5. Applications

5.1 From the viewpoint of both porosity testing and functional significance, it is useful to divide porosity into two broad categories, namely intrinsic porosity and gross defects.<sup>5,6</sup>

5.1.1 *Intrinsic* or *normal* porosity is due primarily to small deviations from ideal plating and surface preparation conditions. As such, it will be present to some degree in all commercial thin platings and will generally follow an inverse relationship with thickness. In addition, scanning electron microscope (SEM) studies have shown that the diameter of such pores at the plating surface is of the order of micrometers, so that only small areas of underlying metal are exposed to the environment.

5.1.2 *Gross defects*, on the other hand, would result in comparatively large areas of exposed basis metal or underplat-

ing. Examples of such defects are mechanical damage to the coating through mishandling or wear. Gross defects can also be found in undamaged coatings in the form of networks of microcracks and as large as-plated pores—with diameters an order of magnitude (or more) greater than intrinsic porosity. Such gross defects indicate such serious deviations from acceptable coating practice as dirty substrates and contaminated or out-of-balance baths.

5.2 Intrinsic porosity and most types of gross defects are too small to be seen except at magnifications so high that a realistic assessment of the overall coating surface in the functional areas of the part cannot be made. Instead, the presence and severity of the porosity is normally determined by some type of pore-corrosion test that will magnify the pore sites by producing visible reaction products in and around the pores or cracks. Tests for gross defects (Section 7), and especially for mechanical damage and wear, are designed to be less severe. Such tests, however, may not detect a sizeable portion of the smaller (intrinsic) pores in a coating. On the other hand, standard tests for intrinsic porosity (Section 6) will easily reveal the presence of gross defects as well.

5.3 Porosity tests are generally destructive in nature and are designed to assess the quality of the coating process in conjunction with the substrate. Therefore, separate test specimens are not ordinarily allowed.

5.4 In the tests summarized in this guide, chemicals react with the exposed substrate through the pore or channel to form a product that is either directly observable or that is made observable by subsequent chemical development.

5.5 Porosity tests differ from corrosion and aging tests. A good porosity test process must clean, depolarize, and activate the substrate metal exposed by the pore, and attack it sufficiently to cause reaction products to fill the pore to the surface of the coating. The corrosive reagent ideally does not react with the coating. Reaction time is limited, particularly with thin coatings, since the corrosive will attack the substrate in all directions and, in so doing, undermine the coatings so that false observations may be made. When the corrosion product is soluble in the reagent, a precipitating indicator is used to form the reaction product.

5.6 The substrate exposed by the pores may be the basis

<sup>5</sup> Baker, R. G., Holden, C. A., and Mendizza, A., Proceedings of the American Electroplaters Society, Vol 50, 1963, p. 61.

<sup>6</sup> Krumbein, S. J., "The ASTM Approach to Porosity Testing," Proc. 1991 International Technical Conf. of the American Electroplaters and Surface Finishers Soc., (SUR/FIN '91), Toronto, 1991, pp. 527-536.



metal, an underplate, or both.

## 6. Outlines of Standard Porosity Tests for Intrinsic Porosity (5.1.1)

NOTE 2—The test methods outlined in this section are only summaries. The specified test standard must be referred to for the instructions on how to perform the tests, as well as for *important applications and limitations*.

### 6.1 Nitric Acid Vapor Test (Test Method B 735):

6.1.1 *Scope*—Gold coatings on nickel, copper, and their alloys, where the gold is at least 0.6  $\mu\text{m}$  (24  $\mu\text{in.}$ ) thick.

6.1.2 *Summary of Test Method*—Test specimens are suspended over concentrated nitric acid in a closed nonreactive vessel for a specified time, usually 0.5 to 2.0 h. After exposure, the specimens are dried in an oven to fix the reaction products. Each reaction product spot indicates a pore in the coating.

### 6.2 Paper Electrography (Test Method B 741):

6.2.1 *Scope*—Gold and palladium coatings on nickel, copper, or copper alloys that have flat or nearly flat surfaces, or, with appropriate fixtures, gently curved surfaces.

6.2.2 *Summary of Test Method*—Electrolyte-soaked paper is pressed against the specimen at controlled pressure. Current, at a constant controlled dc voltage, is passed from an inert cathode, through the paper, to the specimen (which is made the anode) for a specified time. Base-metal ions at the pore sites migrate to the paper where an indicator reagent converts them to colored products (the pore indications).

### 6.3 Sulfur Dioxide Test:

#### 6.3.1 *Scope*:

6.3.1.1 Variation A applies to gold, palladium, and silver over nickel, copper, and their alloys (Test Method B 799).

6.3.1.2 Variation B applies to tin and its alloys over iron or steel (Appendixes of Specification B 545 and Specification B 605).

6.3.2 *Summary of Test Method*—The test specimens are suspended over sulfur-oxide acidic solutions in a sealed chamber. For Variation A, the solution is concentrated sulfurous acid. For Variation B it is a 1:4 mixture of 0.1N sulfuric acid and 0.12N sodium thiosulfate solutions. Each reaction product spot on the surface indicates a pore in the coating.

### 6.4 Humid Sulfur Vapor (“Flowers-of-Sulfur”) (Test Method B 809):

6.4.1 *Scope*—Primarily for coatings over silver, copper, or copper alloys. Coatings may include nickel, gold, palladium, tin, and any other coating that does not significantly tarnish in reduced sulfur atmospheres.

6.4.2 *Summary of Test Method*—Test specimens are suspended in a vented closed container over powdered sulfur at controlled humidity and temperature. Black or brown spots indicate porosity.

### 6.5 Gel Electrography (Test Method B 798):

6.5.1 *Scope*—Gold and palladium coatings over nickel, copper, or copper alloys.

6.5.2 The test sample is made the anode in a cell of a specific geometry, which contains a solid electrolyte consisting of gelatin, conducting salts and an indicator. Application of a constant dc current causes migration of base-metal ions through the pores to the metallic coating surface where reaction with the indicator produces colored reaction products.

### 6.6 Ferroxyl Test (Annexes of Specification B 689, Specifici-

cation B 650, and Specification B 734):

6.6.1 *Scope*—Metallic coatings included are those that are resistant to ferricyanide and chloride, but are cathodic to iron, steel, or iron-based alloy substrates. Examples of such coatings are gold, tin, nickel, copper, chromium, and their alloys.

6.6.2 *Summary of Test Method*—Electrolyte-wetted, gel-chloride treated paper strips are placed firmly in contact with test specimen surfaces for a specified time, not to exceed 10 min. After the allotted time, the paper strips are wetted with a ferricyanide indicator solution. Blue spots indicate pores.

6.6.3 Alternate methods involve formation of the blue spots directly on the specimen (Specification B 733, Test Methods, Ferroxyl Test for Iron Base Substrates).

## 7. Outlines of Porosity Tests for Gross Defects and Mechanical Damage (5.1.2)

NOTE 3—The test methods outlined in this section have been taken from ASTM specifications for specific metallic coatings. As such they are only summaries. ASTM Subcommittee B08.10 is developing complete test method documents for some of those procedures.

### 7.1 Hot Water Test (Annex of Specification B 689):

7.1.1 *Scope*—Metallic coatings cathodic to a ferrous substrate; for example, nickel, tin, or gold on steel or iron-based alloy substrates.

7.1.2 *Summary of Test Method*—The test specimens are immersed for a specified time in neutral, distilled heated (85°C) water that is agitated with clean air. After exposure and drying, black spots and red rust indicate porosity.

7.1.3 Alternative methods involve aerated water at room temperature with longer exposure times.

### 7.2 Alizarin Test (Specification B 733, Test Methods, “Porosity”):

7.2.1 *Scope*—Primarily for nickel on aluminum substrate.

7.2.2 *Summary of Test Method*—The surface of the specimen is swabbed with a sodium hydroxide solution. After rinsing, an alizarin sulfonate solution is applied in two steps followed by glacial acetic acid to remove the background violet color. Any red spots remaining indicate pores.

### 7.3 Sodium Polysulfide Immersion (Specification B 246) is an example):

7.3.1 *Scope*—For detecting gross discontinuities, including wear and mechanical damage, in coatings over copper or its alloys. Applicable coatings include tin, nickel, gold, palladium, or any other coating that does not tarnish in the polysulfide solution.

7.3.2 *Summary of Test Method*—Immerse samples in an alkaline solution of sodium polysulfide for 60 s. After rinsing and drying, samples are examined for dark or discolored areas.

### 7.4 Ferrocyanide Test (Specification B 733, Test Methods, “Porosity”):

7.4.1 *Scope*—For gross defects in nickel coatings over copper substrates.

7.4.2 *Summary of Test Method*—Wipe the coated specimen with glacial acetic acid. After 3 min, apply a solution of potassium ferrocyanide and methyl cellulose in boiling distilled water. The appearance of brown spots after 2 min indicates pores.

### 7.5 Phosphomolybdic Acid (PMA):

 **B 765**

7.5.1 *Scope*—Individual specimens of gold, silver, or palladium, over nickel, copper, or their alloys.

7.5.2 *Summary of Test Method*—The part is exposed briefly to hydrochloric acid fumes. Then a small drop of aqueous PMA solution is applied to the test area. The presence of any exposed substrate metal is revealed by the formation of an intensely colored molybdenum blue complex.

## 8. Report

8.1 The results of a porosity test are usually given in terms of one of the following:

8.1.1 *Option 1*—The number and size of the pore indications in the measurement area. This is converted to a pore *density* in terms of number of pores per square centimetre. The pore indications are often classified according to size (see the individual test standard).

8.1.2 *Option 2*—The percentage of the significant surface covered by the pore indications.

8.1.3 *Option 3*—The area, in square millimetres, of the largest spot on the significant surface.

## 9. Evaluation

9.1 *Procedure*—In Option 1, count individual pores at 10× magnification in the significant area of the coating, as defined by the specification or drawing of the product. In Option 2, compare results with panels shown in Test Method B 276 and Practice B 537, or those supplied as criteria by the purchaser. In Option 3, scan for the largest defect(s).

9.2 *Criteria (Pass-Fail)*—Pass-fail criteria, such as pore count greater than 50/cm<sup>2</sup> (Option 1), or an area greater than 1 % (Option 2), or a spot or crack with a total area greater than 2.5 mm<sup>2</sup> (Option 3), should be chosen in conformance to the intended function of the coating and the intended use of the object coated. Such criteria should be agreed upon between supplier and purchaser.

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*