



Designation: B 679 – 98

# Standard Specification for Electrodeposited Coatings of Palladium for Engineering Use<sup>1</sup>

This standard is issued under the fixed designation B 679; 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 specification covers requirements for electrodeposited palladium coatings containing at least 99.7 mass % of palladium metal. Composite coatings consisting of palladium with a thin gold overplate for applications involving electrical contacts are also covered.

1.2 *Properties*—Palladium is the lightest and least noble of the platinum group metals. It has a specific gravity of 12.0, which is substantially less than gold (19.3) and platinum (21.5). This yields a greater volume or thickness of coating and, consequently, some saving of metal weight accompanied by a small sacrifice in corrosion resistance and reflectivity. The following table compares the hardness range of electrodeposited palladium with other electrodeposited noble metals and alloys (1,2).<sup>2</sup>

	Approximate Hardness (HK <sub>25</sub> )
Gold	50–250
Palladium	75–600
Platinum	150–550
Palladium-Nickel	300–650
Rhodium	750–1100
Ruthenium	600–1300

1.3 The values stated in SI units are the preferred values. Values provided in parentheses are for information only.

1.4 *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 The following standards form a part of this specification to the extent referenced herein:

### 2.2 ASTM Standards:

B 242 Practice for Preparation of High-Carbon Steel for Electroplating<sup>3</sup>

- B 254 Practice for Preparation of and Electroplating on Stainless Steel<sup>3</sup>
- B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings<sup>3</sup>
- B 322 Practice for Cleaning Metals Prior to Electroplating<sup>3</sup>
- B 374 Terminology Relating to Electroplating<sup>3</sup>
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section<sup>3</sup>
- B 488 Specification for Electrodeposited Coatings of Gold for Engineering Use<sup>3</sup>
- B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals<sup>3</sup>
- B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals<sup>3</sup>
- B 507 Practice for Design of Articles to Be Electroplated on Racks<sup>3</sup>
- B 542 Terminology Relating to Electrical Contacts and Their Use<sup>4</sup>
- B 558 Practice for Preparation of Nickel Alloys for Electroplating<sup>3</sup>
- B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method<sup>3</sup>
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry<sup>3</sup>
- B 571 Test Methods for Adhesion of Metallic Coatings<sup>3</sup>
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings<sup>3</sup>
- B 689 Specification for Electroplated Engineering Nickel Coatings<sup>3</sup>
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings<sup>3</sup>
- B 741 Test Methods for Porosity in Gold Coatings on Metal Substrates by Paper Electrography<sup>3</sup>
- B 748 Test Method for Measurement of Thickness of Metallic Coatings by Measurement of a Cross-Section with a Scanning Electron Microscope<sup>3</sup>
- B 762 Method of Variables Sampling of Metallic and Inorganic Coatings<sup>3</sup>

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.02 on Precious Metal Coatings.

Current edition approved March 10, 1998. Published November 1998. Last previous edition B 679 – 80. Originally published as B 679 – 91.

<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this specification.

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

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.04.

- 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>
- D 3951 Standard Practice for Commercial Packaging<sup>4</sup>

**3. Terminology**

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *underplating*—a metallic coating layer between the basis metal or substrate and the topmost metallic coating. The thickness of an underplating is usually greater than 1 μm (40 μin.), in contrast to a strike or flash.

**4. Classification**

4.1 Orders for articles to be plated in accordance with this specification shall specify the plating system, indicating the basis metal, the thickness of the underplatings, the thickness of the palladium coating, and the grade of the gold overplating according to Tables 1 and 2.

**5. Ordering Information**

5.1 In order to make the application of this standard complete, the purchaser needs to supply the following information to the seller in the purchase order or other governing document:

- 5.1.1 The name, designation, and date of issue of this standard.
- 5.1.2 The coating system including basis metal, thickness class and gold overplate grade (see 4.1 and Tables 1 and 2).
- 5.1.3 Presence, type, and thickness of underplating (see 3.2.3).
- 5.1.4 Significant surfaces shall be defined (see 3.2.2).
- 5.1.5 Requirements, if any, for porosity testing (see 9.5):
- 5.1.6 Requirement, if any, for bend ductility testing (see 9.6):
- 5.1.7 Sampling plan employed (see Section 8), and
- 5.1.8 Requirement, if any, for surface coating cleanliness (absence of residual salts). See Appendix X3.

**6. Manufacture**

6.1 Any process that provides an electrodeposit capable of meeting the specified requirements will be acceptable.

6.2 *Substrate:*

**TABLE 2 Gold Overplate<sup>A</sup>**

Grade	Type	MIL-G-45204	Hardness (Code)	Thickness Range
0	No Overplate	...	...	...
1	1 (99.9 % Au min)	III	90 HK <sub>25</sub> max (A)	0.05-0.12 μm
2	2 (99.7 % Au min)	I	130-200 HK <sub>25</sub> (C)	0.05-0.25 μm

<sup>A</sup>See Specification B 488 and Appendix X1

6.2.1 The surface condition of the basis metal should be specified and should meet this specification prior to the plating of the parts.

6.2.2 Defects in the surface of the basis metal, such as scratches, porosity, pits, inclusions, roll and die marks, laps, cracks, burrs, cold shuts, and roughness may adversely affect the appearance and performance of the deposit, despite the observance of the best plating practice. Any such defects on significant surfaces should be brought to the attention of the supplier and the purchaser.

6.2.3 Clean the basis metal as necessary to ensure a satisfactory surface for subsequent electroplating in accordance with Practices B 183, B 242, B 254, B 281, B 322, B 343, B 481, B 482 and B 558.

6.2.4 Proper preparatory procedures and thorough cleaning of the basis metal are essential for satisfactory adhesion and performance of these coatings. The surface must be chemically clean and continuously conductive, that is, without inclusions or other contaminants. The coatings must be smooth and as free of scratches, gouges, nicks, and similar imperfections as possible.

NOTE 1—A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these may not be normal in the treatment steps preceding the plating, and a special agreement is indicated.

6.3 Apply the coating after all basis metal preparatory treatments and mechanical operations on significant surfaces have been completed.

6.4 *Racking:*

6.4.1 Position parts to allow free circulation of solution over all surfaces. The location of rack or wire marks in the coating should be agreed upon between the producer and supplier.

6.5 *Plating Process:*

6.5.1 *Nickel Underplating*—Apply a nickel underplating before the palladium when the product is made from copper or copper alloy. Nickel underplatings are also applied for other reasons. See Appendix X2.

NOTE 2—In certain instances where high frequency analog signals are employed, such as wave guides, the magnetic properties of nickel may attenuate the signal. Palladium itself is non-ferromagnetic.

6.5.2 *Strikes*—Standard practice calls for a gold or palladium strike to follow any underplate or substrate (other than silver or platinum) immediately prior to applying the palladium.

6.5.3 *Plating*—Good practice calls for the work to be electrically connected when entering the bath. A minimum of 0.5 V is suggested. During electroplating it is extremely important to maintain the voltage, current density, or both beneath the value for hydrogen evolution. (See 7.2)

**TABLE 1 Thickness Class<sup>A</sup>**

Thickness Class	Minimum Thickness of Pd (μm)
0.08	0.08
0.15	0.15
0.25	0.25
0.50	0.50
0.75	0.75
1.00	1.00
1.25	1.25
1.5	1.5
2.5	2.5
3.0	3.0
5.0	5.0

<sup>A</sup>See Appendix X4 for specific applications of the various thickness classes.

6.5.4 *Stress Cracking*—Problems associated with the incorporation of hydrogen in the palladium, which can lead to stress cracking of the coating, shall be controlled by choosing plating baths and plating conditions that minimize the H/Pd deposition ratio. (3) The presence of stress-induced microcracks that penetrate to the underlying substrate or underplating can be detected with one of the porosity tests specified in 9.5.

6.5.5 *Gold Overplating*—Apply a thin gold overplating after the palladium in any application in which palladium plated electrical connectors are mated together in a contact pair. This process is necessary to preserve the performance of the contact surface. See Appendix X1 for other reasons for using a gold overplate.

NOTE 3—When using Type 1 gold, the thickness of the gold overplate shall not exceed 0.12  $\mu\text{m}$  (5  $\mu\text{in.}$ ) due to increased risk of degrading durability and increasing the coefficient of friction.

6.5.6 *Residual Salts*—For rack and barrel plating applications, residual plating salts can be removed from the articles by a clean, hot (50 to 100°C) water rinse. A minimum rinse time of 2.5 min (racks) or 5 min (barrel) is suggested. Best practice calls for a minimum of three dragout rinses and one running rinse with dwell times of 40 s in each station when rack plating and 80 s when barrel plating. Modern high-velocity impingement type rinses can reduce this time to a few seconds. This is particularly useful in automatic reel-to-reel applications where dwell times are significantly reduced. See Appendix X3.

## 7. Coating Requirements

7.1 *Nature of Coating*—The palladium deposit shall have minimum purity of 99.7 mass %.

7.2 *Appearance*—Palladium coatings shall be coherent, continuous, and have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

7.3 *Thickness*—Everywhere on the significant surface (see 5.1.4), the thickness of the palladium coating shall be equal to or exceed the specified thickness. The maximum thickness, however, shall not exceed the drawing tolerance.

NOTE 4—The coating thickness requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces while conforming to all maximum thickness tolerances given in the engineering drawing. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the plating process.

In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement.

7.4 *Adhesion*—The palladium coatings shall be adherent to the substrate, when tested by one of the procedures summarized in 9.4.

7.5 *Integrity of the Coating*:

7.5.1 *Gross Defects/Mechanical Damage*—The coatings shall be free of visible mechanical damage and similar gross defects when viewed at magnifications up to 10 $\times$ . For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside of or on the periphery of the significant surfaces. See 7.5.2 and 6.5.4.

7.5.2 *Porosity*—Almost all as-plated electrodeposits contain some porosity, and the amount of porosity to be expected for any one type of coating will increase with decreasing the thickness of that particular coating type. The amount of porosity in the coating that may be tolerable depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number, or away from the significant surfaces, their presence can often be tolerated. Acceptance or pass-fail criteria, if required, shall be part of the product specification for the particular article or coating requiring the porosity test. See 9.5.

NOTE 5—Extensive reviews of porosity and porosity testing can be found in the literature (4, 5)

## 8. Sampling

8.1 The sampling plan used for the inspection of a quality of the coated articles shall be as agreed upon between the purchaser and the supplier.

NOTE 6—Usually, when a collection of coated articles, the inspection lot (see 8.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles—the sample—is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 identifies the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

## 9. Test Methods

9.1 *Deposit Purity*—Use any recognized method to determine qualitatively the impurities present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the

metallic impurities. Initial scanning should be carried out for all elements, in order to detect any unknown or unexpected impurities. Determine deposit purity by subtracting total impurities from 100 %.

**NOTE 7**—Deposit purity is best determined on a special test specimen. One must be careful to arrange the specimen so as to electroplate at a typical density, similar to the production pieces. Palladium may be stripped by utilizing a 90 volume % (reagent grade) sulfuric, 10 % (reagent grade) nitric acid solution. The test specimen substrate should be platinum, gold, or an electrodeposit not attacked by the strip solution. The total palladium deposit should be over 100 mg and the sample weight is determined by a weigh-strip-weigh procedure. The strip solution is then used for quantitative analysis of impurities.

**9.2 Appearance**—The coating shall be examined at up to 10× magnification for conformance to the requirements of appearance.

**9.3 Thickness**—Measure thickness by methods outlined in Test Methods B 487, B 499, B 567, B 568, or B 748, or any other test method that has an uncertainty less than 10 %, or less than the test methods listed.

**9.4 Adhesion**—Determine adhesion by one of the following procedures (see Test Methods B 571 for full details):

**9.4.1 Bend Test**—Bend the electroplated article repeatedly through an angle of 180° on a diameter equal to the thickness of the article until fracture of the basis metal occurs. Examine the fracture at a magnification of 10×. Cracking without separation does not indicate poor adhesion unless the coating can be peeled back with a sharp instrument.

**9.4.2 Heat Test**—No flaking, blistering, or peeling shall be apparent at a magnification of 10× after the palladium-electroplated parts are heated to 300 to 350°C (570 to 660°F) for 30 min and allowed to cool.

**9.4.3 Cutting Test**—Make a cut with a sharp instrument and then probe with a sharp point and examine at a magnification of 10×. No separation of the coating from the substrate shall occur.

**9.5 Plating Integrity**—Porosity and microcracks shall be determined by either Test Methods B 741, B 799, or B 809

unless otherwise specified. Do not use the nitric acid vapor test (palladium can dissolve in nitric acid.)

Note the nature of the basis metal, the nature and thickness of any intermediate layers or underplate, and the shape of the palladium plated part. Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits of palladium alloys.

**9.6 Ductility**—When required, determine ductility in accordance with Practice B 489.

## 10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the U.S. Government or an agent of the U.S. Government.

10.1.1 **Sampling**—For government acceptance, the sampling plane specified in MIL-STD-105 is to be used instead of the ASTM standards specified in 8.1.

10.1.2 **Thickness Testing:**

10.1.2.1 In addition to the non-destructive methods outlined in Practice B 499 and Test Methods B 567 and B 568, a cross-sectioning method, such as that specified by Test Method B 487 or B 748, shall be used as a referee method to confirm the precision and bias of the particular non-destructive technique that is used.

10.1.2.2 The palladium thickness on significant surfaces shall be at least 1.3 μm (0.05 mil), *unless otherwise specified on the drawings or in the contract*. The coating on nonsignificant surfaces shall be of sufficient thickness to ensure plating continuity and uniform utility, appearance, and protection. The thickness of plating on nonsignificant surfaces, unless specifically exempted, shall be a minimum of 60 % of that specified for significant surfaces.

10.1.3 **Packaging**—The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.

**NOTE 8—Caution:** Some contemporary packaging materials may emit fumes that are deleterious to the coating surface.

## APPENDIXES

### (Nonmandatory Information)

#### X1. SOME REASONS FOR USING A GOLD OVERPLATE

X1.1 A gold overplate is employed to enhance the performance of the palladium surface. Two types of gold are used:

X1.1.1 Type 1 gold is used in the critical areas in thickness ranges of 0.05 to 0.12 μm.

X1.1.2 Type 2 gold is used in the critical areas in thickness ranges of 0.05 to 0.25 μm or higher.

X1.2 The gold overplate offers the following performance enhancements to palladium:

X1.2.1 **Durability**—A gold overplate of proper thickness can reduce friction and enhance durability by providing a low shear-strength solid lubricant that reduces friction and wear

(6,7). Type 1 gold should be used at a thickness no greater than 0.12 μm to maintain a low coefficient of friction. Palladium-should not be mated against itself in a sliding contact pair when durability and resistance to fretting and frictional polymer formation is desired.

X1.2.2 **Mating Force**—Application of Type 1 or Type 2 gold can reduce friction and mating force. Type 1 should be no more than 0.12 μm thick.

X1.2.3 **Fretting**—Fretting occurs when two surfaces undergo low amplitude, repetitive motions. Depending on conditions and contact surface materials, fretting wear or fretting corrosion can occur. Fretting wear is loss of material along the

wear track. Fretting corrosion is the formation of surface oxides at the contact surface. The addition of a Type 1 or Type 2 gold can often reduce fretting corrosion that is due to fretting motions (8). The occurrence of fretting is influenced greatly by contact design. See Terminology B 542.

X1.2.4 *Frictional Polymerization*—Frictional polymerization is the formation of insulating polymeric films at the

contact spot. Such occurrences have been documented for palladium, palladium-nickel alloys and other metals (7). The addition of a Type 1 or Type 2 gold overplate can often reduce frictional polymer formation (8). (See Terminology B 542)

## X2. SOME REASONS FOR USING A NICKEL UNDERPLATE FOR PALLADIUM-NICKEL ELECTROPLATING

X2.1 *Diffusion Barrier*—To inhibit diffusion of copper from the basis metal into the palladium.

X2.2 *Levelling Layer*—To produce a smoother surface than the basis metal in order to ensure a lower porosity palladium top coat, for example, levelling nickel over a rough substrate.

X2.3 *Pore Corrosion Inhibitor*—A nickel underplate under the palladium top coat will form passive oxides at the base of pores in humid air, provided the environment does not contain significant amounts of acidic pollutants, such as SO<sub>2</sub> or HCl.

X2.4 *Load Bearing Underlayer for Contacting Surfaces*—A hard nickel underplate can serve as a load bearing foundation for the palladium top coat and reduce the wear of the precious metal during sliding of the contacting surfaces.

X2.5 For all of these purposes, the nickel underplating must be intact, that is, not cracked, and must have sufficient thickness to achieve the particular function for which it was intended. As a general rule, the minimum thickness should be 1.3 μm (50 μin.), preferably greater. For some levelling purposes, a greater thickness may be required.

## X3. RESIDUAL SALTS

X3.1 Electroplated parts are placed in water of known conductivity and agitated for a specific time. The conductivity of the water extract is measured and the increase in conductivity due to residual salts and other conducting impurities is calculated. A suggested water extract conductivity test method uses a procedure in accordance with Test Methods D 1125, Method A.

X3.2 Conductivity of water for extract test shall be 1 μS/cm or less (resistivity 1 MΩ·cm or more).

X3.2.1 A sample of the coated parts having a total surface area of 30 cm<sup>2</sup> shall ordinarily be used and extracted in 100 cm<sup>3</sup> of equilibrated water. To prepare equilibrated water, fill a clean polyethylene bottle half-way with high-purity water (X6.1), replace the bottle cap and shake the bottle vigorously for 2 min to equilibrate the water with the CO<sub>2</sub> in the air. CO<sub>2</sub> is a component of air, is soluble in water, and forms carbonic acid, which ionizes and is at equilibrium at 0.8 μS/cm. Slowly

agitate the solution for 10 min before determining the conductivity of the extract. In a closed polyethylene bottle, the equilibrated water will remain in the range from 0.8 to 1 μS/cm for at least 1 week.

X3.3 Inspection under a source of ultraviolet light is often employed to determine whether electroplating salts have been removed by the rinsing following gold electroplating. The presence of salts is evidenced by a characteristic fluorescence and should not be confused with fluorescing dirt or dirt particles.

X3.4 Water or purging stains, resulting from blind holes or from parts that were assembled before electroplating, as normally obtained in good commercial practice, are permissible except where they occur on surfaces to which electrical contact is to be made or on which subsequent soldering operations are performed.

## X4. RECOMMENDED THICKNESSES

X4.1 Palladium thicknesses that have been recommended for specific applications are given in the following table.

Thickness, μm	Application
0.08–0.25	Semiconductor Lead Frames in Integrated Circuitry (9). Also solderable surfaces on Printed Wiring Boards
0.25–0.5	Catalysts. Also electrical contacts where little adverse environmental, electrical, or mechanical action is expected.
0.75–1.5	Low-energy electrical connector contacts.
2.5–5	Relay contacts with mechanical and electrical erosion.

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