



# IPC-TM-650 TEST METHODS MANUAL

**1.0 Outline for Methods** The part is exposed to an atmosphere that is corrosive to the basis metal. Where there is porosity, the reagent attacks the basis metal and generates corrosion products. The part is examined for corrosion products.

**1.1 Method 1 (Nitric Acid Vapor-Gold on Copper)** This method applies only to gold coatings on copper and copper-base alloys.

**1.2 Method 2 (Extended Nitric Acid Vapor)** This method applies only to gold coatings on copper and copper-base alloys.

**1.3 Method 3 (Nitric Acid Vapor - Gold on Nickel)** This method applies to gold coatings on copper, copper-base alloys, and nickel.

## 2.0 Nitric Acid Vapor - Gold on Copper

**2.1 Apparatus** Methods 1, 2, and 3.

**2.1.1 Collimated Incandescent Lamp** No. 31-33-53, Bausch and Lomb Co., or No. 359, American Optical Co., or equivalent.

**2.1.2 Desiccator (Glass)** Fisher Scientific Co. Series 8-595 or 8-624 or equivalent. The size of the desiccator shall be chosen so that no more than 164 cm<sup>3</sup> of air space exists for 6.45 cm<sup>2</sup> of nitric acid surface area when approximately 301 ml of acid are placed in the bottom.

*Caution: No sealant (e.g., stopcock grease, etc.) shall be applied to the mating surfaces of the desiccator cover or bottom. Sealants may cause these to stick together, and any method employed to release a stuck cover is likely to be extremely hazardous.*

**2.1.3 Part Support** A supporting structure made of glass or other material not attached by nitric acid vapors to hold the parts under test in the upper part of the desiccator.

**2.1.4 Oven** An oven capable of drying parts at 125°C.

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Subject <b>Porosity of Metallic Coatings on Copper-Base Alloys and Nickel (Nitric Acid Vapor Test)</b>	
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Originating Task Group <b>N/A</b>	

**2.1.5 Pressure Sensitive Polytetrafluoroethylene Tape with Silicone Adhesive Backing** Connecticut Hard Rubber Company TFE Type HM225 or equivalent.

**2.2 Reagents** Methods 1 and 2. Nitric Acid, 69, 0 to 71.0 Percent HNO<sub>3</sub>.

## 3.0 Procedure

### 3.1 Method 1 (Nitric Acid Vapor-Gold on Copper)

*Caution: Perform all work in hood, since the vapors given off are toxic. Chemical goggles, completely closing the eyes, should be worn and eyewash facilities should be readily available.*

To minimize and tendency for the cover to stick, press a minimum of three strips of the pressure polytetrafluoroethylene tape (adhesive side down) at equal intervals around the mating surface of the bottom of the desiccator. Place approximately 300 ml of nitric acid in the bottom of the desiccator. Cover the desiccator and allow about 30 minutes for equilibrium to be established before starting the test. This equilibrium is necessary only when the nitric acid is first placed in the desiccator.

Clean the part with 1,1,1-trichloroethane or toluene or other suitable solvent and dry with filtered dry air (gage pressure less than 207 kPa (30 psi)).

Inspect the cleaned part at 10 power magnification for evidence or particulate matter of the surface. If any remains, reclean the solvent or use a clean soft brush to remove it prior to the start of the porosity test.

Place the clean part on the support so that adequate space exists to allow circulation of acid vapor and air around it.

Carefully remove the desiccator cover, place the support in the desiccator and immediately replace the cover to prevent an excessive loss of vapors that would disrupt the equilibrium previously established. The test shall be performed at a temperature of 24 ± 3°C and a maximum relative humidity of 60 percent. Unless otherwise specified, the time of exposure to the reagent vapor shall be one hour.

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Remove the part from the desiccator and immediately transfer it to an oven at 125°C for 30 minutes. Remove and inspect the part with 10 power magnification using the collimated incandescent light source arranged so that the light beam strikes the surface being examined at an angle of less than 15 degrees. The presence of blue, green, or bluish-white protrusions indicates that the coating is discontinuous (i.e., porous, scratched, etc.) at these spots. The acceptable number, sizes, and location of these protrusions shall be as specified on the appropriate drawing or specification.

### 3.2 Method 2 (Extended Nitric Acid Vapor-Gold on Copper)

**Procedure** This method is an extension of method 1 and may be used only after the test specimen has passed the requirements of the visual examination with the collimated light source. The method permits the determination of the actual sizes of the larger pores. The inspection should be performed as indicated below.

After examining the oven-dried test specimen in accordance with Method 1, brush the corrosion products from the specimen. Inspect the area where the corrosion products appeared at a magnification that permits measurements of the largest dimensions of the discontinuities with reasonable accuracy. The acceptable distribution of pore sizes shall be as specified on the appropriate drawing or specification.

### 3.3 Method 3 (Nitric Acid Vapor-Gold on Nickel)

**3.3.1 Apparatus** The apparatus described in methods 1 and 2.

**3.3.2 Reagents** Nitric acid, 69.0 to 71.0 percent HNO<sub>3</sub>. Sodium Hydroxide 10 percent by weight solution. Polysulfide reagent.

**3.3.3 Procedure** *Caution: Perform all work in a hood, since vapors given off are toxic. Chemical goggles, completely enclosing the eyes, should be worn and eye wash facilities should be readily available.*

Use the procedure for Method 1 up to removal from nitric acid vapors.

At the end of the one hour exposure to the nitric acid vapors, remove the part from the desiccator and immediately dip it in the 10 percent by weight sodium hydroxide solution for 25 to 30 seconds at room temperature. Rinse the part with water and then dip it for 25 to 30 seconds in the polysulfide reagent at room temperature. Then rinse it in water and dry using filtered compressed air (gage pressure less than 207 kPa).

Inspect the part at 10 power magnification. The presence of black corrosion products is evidence of porosity. The acceptable number, sizes, and locations of pores shall be as specified on the appropriate drawing or specification.

**3.4 Polysulfide Reagent Makeup** Dissolve sodium sulfide crystals in water until solution is saturated. Add excess flowers of sulfur (more than 250 grams per 1000 ml). Stir and allow solution to stand 24 hours. Filter and dilute with water to a specific gravity of 1.142 at room temperature as defined in ASTM Designation E 41. The solution should be a red-orange color.

To check reagent effectiveness, dip a clean copper or high (more than 95 percent) copper alloy specimen into the reagent at room temperature as defined in ASTM Designation E 41. It shall blacken within 10 seconds.