



IPC-TM-650 TEST METHODS MANUAL

1 Scope This is an electrogravimetric method for determining the purity of copper foil or plating.

2 Applicable Documents None

3 Test Specimens Each copper specimen must weigh approximately five grams and be raw copper foil or plating without treatment.

4 Apparatus or Material

4.1 Electrodes for Electrogravimetric Analysis A platinum gauze cathode. A platinum gauze rotating anode or a spiral platinum wire anode.

4.2 Reagents

4.2.1 Acid mixture for dissolving sample: Add 300 ml of concentrated H₂SO₄ slowly with stirring to 750 ml of water. Cool and add 210 ml of HNO₃.

4.2.2 For cleaning the sample use 5% H₂SO₄. For cleaning of the interior of the glassware use diluted HNO₃ (one part acid plus three parts distilled or deionized water by volume).

4.2.3 For final rinsing use absolute methanol or equivalent.

4.2.4 Distilled or deionized water.

4.3 Other apparatus

4.3.1 Fume hood for removing fumes from dissolution.

4.3.2 Hot plate for heating the test solution to 80-90 °C [176-194 °F].

4.3.3 Analytical balance capable of weighing copper sample and platinum cathode to the nearest 0.1 mg.

4.3.4 Oven for drying the specimen and cathode at approximately 110 °C [230 °F].

4.3.5 Current source capable of supplying a current density based on the cathode area of at least 0.6 A/dm² [6 ASF].

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4.3.6 Tall-form 180 ml to 300 ml lipless beaker provided with a close-fitting split cover.

5 Procedure

5.1 Test

5.1.1 Clean the copper foil by dipping in 5% H₂SO₄ at room temperature, wash thoroughly in tap water, then distilled water, rinse in absolute methanol or equivalent, dry for a few minutes in hot air oven at 110 °C [230 °F] and cool in a desiccator. Weigh the copper test specimen to the nearest 0.1 mg and transfer to a tall-form 180 ml to 300 ml lipless beaker provided with a close-fitting cover. Place the beaker into a fume hood.

5.1.2 Add 45 ml of H₂SO₄-HNO₃ mixture and allow to stand covered for a few minutes until reaction has nearly ceased.

5.1.3 Heat at a temperature of 80-90 °C [176-194 °F] until dissolution is complete and brown fumes have been expelled. Never boil.

5.1.4 Cool slightly and carefully wash down the cover and insides of the beaker with distilled water and dilute the solution sufficiently to cover the cathode cylinder. The purpose of the wash is to make sure that any of the ionized copper that may be on the cover or inside surface of the beaker is in the solution from which the copper is to be reduced by electroplating onto the platinum cathode.

5.1.5 Allow solution to cool to ambient conditions.

5.1.6 Weigh the cathode to the nearest 0.1 mg.

5.1.7 Insert the electrodes in the solution, cover to prevent splashing or evaporation, and electrolyze at a current density of 0.6 A/dm² [6 ASF]. (When a current density of 0.6 A/dm² is used, the electrolysis takes about 16 hours and is conveniently carried on overnight.)

5.1.8 When the solution becomes colorless, reduce the current density to about 0.3 A/dm² [3 ASF] and wash down the cover glasses, electrode stems and the inside of the beaker.

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5.1.9 Continue the electrolysis until the deposition of the copper is complete, as indicated by failure to plate on a new surface of the electrode stem when the level of the solution is raised by adding distilled water. (Metal added to solution by washing glass and cover will typically plate in approximately one hour.)

5.1.10 Without interrupting the current, remove the electrodes from the solution.

5.1.11 Remove the cathode. Wash with distilled water. Dip in absolute methanol or equivalent and dry rapidly in a hot air oven at 110 °C [230 °F] (approximately five minutes). Cool to ambient in a desiccator and weigh to the nearest 0.1 mg.

5.1.12 Shut off current supply to just set up.

5.2 Calculations

5.2.1 Calculate the percentage of copper by weight by the following method:

Weight of copper recovered $C_R = A - B$

where:

A = weight of cathode plus deposited copper in grams.

B = initial weight of the cathode in grams

Copper, by weight (%) = $(C_R/C_i) \times 100$

where:

C_R = weight of copper recovered in grams

C_i = initial weight of copper test sample in grams

6 Notes

6.1 Interferences In this method any silver present in the test sample is deposited with the copper, and is reported as copper.