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**Metallic coatings — Test methods for electrodeposited
gold and gold alloy coatings —
Part 3 : Electrographic tests for porosity**

Revêtements métalliques — Méthodes d'essai des dépôts électrolytiques d'or et d'alliages d'or — Partie 3 : Détermination électrographique de la porosité

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Foreword

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Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings —

Part 3 : Electrographic tests for porosity

1 Scope and field of application

This part of ISO 4524 specifies five electrographic tests for assessing the porosity of electrodeposited gold and gold alloy coatings for engineering, and decorative and protective purposes.

2 Cadmium sulfide paper test

2.1 Applicability

This method is suitable for the examination of gold coatings on copper.

2.2 Materials

During the test, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

2.2.1 Cadmium sulfide paper.

Use filter or duplicating paper of adequate wet strength, with a texture that will produce sharp and uniform electrograms. Soak the paper for 10 min in a fresh 10 % (*m/m*) solution of cadmium chloride hemipentahydrate ($\text{CdCl}_2 \cdot 2,5 \text{H}_2\text{O}$) containing 0,1 % (*V/V*) of hydrochloric acid (HCl , ρ 1,16 to 1,18 g/ml). Remove the excess solution with blotting paper.

Allow the paper to dry partially and then immerse it in a fresh 50 g/l solution of sodium sulfide (Na_2S) for 30 s, after which time the paper should be a uniform yellow colour (indicating complete precipitation of cadmium sulfide, CdS). Wash the paper in running water for approximately 1 h, then hang it up to dry.

2.2.2 Moistened blotting paper.

Soak a good quality white blotting paper in water and dry it to a degree that consistently produces sharply defined electrograms.

2.3 Procedure

Lightly brush the electroplated coating to remove loose dust and debris, then degrease it in 1,1,1-trichloroethane vapour or other suitable solvent.

Place a piece of the cadmium sulfide paper on the electroplated specimen (which acts as the anode). On the other face of the cadmium sulfide paper, place a piece of the moistened blotting paper (2.2.2), followed by a high purity clean aluminium or stainless steel platen (which acts as the cathode). Compress the assembly so that the pressure between the cadmium sulfide paper and the specimen is uniform and between 1,4 and 1,7 MPa. While under compression, pass a smooth ripple-free d.c. current from a source not exceeding 12 V. Set the current density initially at 7,5 mA/cm² of anode area and pass for 30 s.

Allow the electrogram produced on the cadmium sulfide paper to dry. The presence of any defect in the electroplated coating is revealed by a corresponding brown stain on the paper.

NOTE — If an overall black stain is obtained in this test, either the electrolyte content of the papers or the current density is too high.

3 Nioxime paper test

3.1 Applicability

This method is suitable for the examination of gold coatings on undercoats of nickel or tin-nickel alloy.

3.2 Materials

During the test, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Nioxime paper.

Soak filter or duplicating paper for 10 min in an 8 g/l solution of nioxime (cyclohexan-1,2-dione dioxime).

Remove the excess solution by blotting and hang the paper up to dry.

3.3 Procedure

Lightly brush the electroplated coating to remove loose dust and debris, then degrease it in 1,1,1-trichloroethane vapour or other suitable solvent.

Moisten a piece of the nioxime paper (3.2) with water. Remove excess water by blotting. Place the treated nioxime paper on the electroplated specimen (which acts as the anode). On the other face of the nioxime paper, place a piece of the moistened blotting paper (2.2.2), followed by a high purity clean aluminium or stainless steel platen (which acts as the cathode). Compress the assembly so that the pressure between the nioxime paper and the specimen is uniform and between 1,4 and 1,7 MPa. While under compression, pass a smooth ripple-free d.c. current from a source not exceeding 12 V. Set the current density initially at 7,5 mA/cm² of anode area and pass for 30 s.

Expose the electrogram produced on the nioxime paper to ammonia vapour and then allow to dry. The presence of any defect in the electroplated coating is revealed by a corresponding pink stain on the paper. When electroplated on copper, defects in the nickel or tin-nickel undercoat are revealed as green stains.

4 Dye-transfer paper test 1 (alternative to the test described in clause 2)

4.1 Materials

During the test, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Dye-transfer paper.

Soak a piece of dye-transfer paper for 30 min in a freshly prepared solution containing 0,01 mol/l of sodium chloride (NaCl) and 0,01 mol/l of sodium carbonate (Na₂CO₃) made by dissolving 0,58 g of sodium chloride and 1,06 g sodium carbonate together in 1 litre of water. Remove the excess solution with blotting paper.

NOTE — Dye-transfer paper may be obtained from some suppliers of photographic materials.

4.2 Procedure

Remove loose dirt and debris from the electroplated coating with a soft brush and then degrease it in 1,1,1-trichloroethane vapour or other suitable solvent.

Place a piece of the damp dye-transfer paper (4.1) emulsion side down on the electroplated specimen (which acts as the anode), followed by a high purity clean aluminium or stainless steel platen (which acts as the cathode). Compress the assembly so that the pressure between the dye-transfer paper and the specimen is uniform and between 1,4 and 1,7 MPa. While under compression, apply a fixed potential of 4 V d.c. for 30 s. Remove the dye-transfer paper and develop it in a saturated ethanolic solution of dithiooxamide (dissolve 0,25 g of dithiooxamide in 100 ml of ethanol by gentle warming; if necessary, filter when cold before use) for 30 s.

Wash the electrogram produced in cold running water and allow to dry. The presence of any defect in the plated coating is revealed by a corresponding dark olive-green stain on the paper.

It is essential that the test papers produced in carrying out the test be rinsed in hot water and carefully dried, on completion of the tests.

NOTE — It is advisable to use tweezers for immersing the paper in the dithiooxamide solution because it can produce persistent black stains on the fingers.

5 Dye-transfer paper test 2 (alternative to the test described in clause 3)

Follow the procedure described in clause 4 but use a developing solution consisting of a 0,5 % (m/m) ethanolic solution of nioxime (cyclohexan-1,2-dione dioxime).

The presence of any defect in the electroplated coating is revealed by a corresponding pink stain on the paper. When plated on copper, defects in the nickel or tin-nickel undercoat are revealed as green stains.

It is essential that the test papers produced in carrying out the test be rinsed in hot water and carefully dried, on completion of the tests.

NOTES (on tests given in clauses 2 to 5)

1 The tests described in clauses 2 and 3 do not call for an electrolyte to be used and rely on ions from the test paper and backing paper to provide conductivity. In some cases, it is necessary to soak the test papers in a solution of a suitable electrolyte, for example for dye-transfer paper (see clause 4) in a 0,1 mol/l solution of sodium nitrate (NaNO₃).

2 The tests described in clauses 4 and 5 are more sensitive than those given in clauses 2 and 3, i.e. microporosity is more easily detected.

6 Electrographic gelatine film test

6.1 Principle

Determination of the porosity of different metal coatings on different basis metals or intermediate metallic layers by means of an electrolytic process in an electrolyte thickened with gelatine and containing a suitable indicator, with which ions from the basis metal form coloured reaction products.

The method may be considered as a variant of electrography which usually is carried out with the aid of a paper soaked in a special test solution. Compared with electrography, this method has some advantages; it can be used also on curved surfaces, and the coloured spots do not spread from the pores to the same extent.

6.2 Reagents

During the test, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.2.1 Gelatine.

Prepare a 10 % (m/m) solution of gelatine at 60 °C with careful stirring to ensure that no air enters the solution.

Store the gelatine solution in a cold place.

6.2.2 Electrolyte solution.

Prepare by mixing 20 ml of ammonia solution ($\rho = 0,88$ g/ml) with 80 ml of water.

6.2.3 Dimethylglyoxime, indicator solution.

Prepare a saturated solution of dimethylglyoxime in ethanol.

6.3 Testing solution

Add, whilst stirring, 35 ml of the electrolyte solution (6.2.2) and 20 ml of the indicator solution (6.2.3) to 950 g of gelatine solution (6.2.1) at 27 to 30 °C, taking care to avoid entrainment of air. Pour the mixture into the electrolysis vessel and leave to cool to the working temperature, 22 to 23 °C.

NOTE — Owing to variations in the quality of the gelatine, no exact working temperature can be indicated. The temperature is, however, suitable for the purposes of this test if a metal sheet which has been dipped in the test solution retains, on removal from the solution, a coating 1 mm thick.

6.4 Apparatus

6.4.1 Water-bath, fitted with a thermostat for indirect temperature control of the test solution.

6.4.2 Stirring device.

6.4.3 Inner tank, for electrolysis in the test solution. The tank shall be made of glass or acid-proof steel. The electrodes (cathodes) shall be made of platinum gauze or gold and have at least the same area as the part to be tested (the anode). The cathodes shall be placed on opposing sides in the test vessel and shall be isolated from the vessel. The electrode distance between the anode and each of the cathodes shall be approximately 50 mm.

6.4.4 Rectifier or accumulator.

6.5 Procedure

6.5.1 Electrolytic process

Immerse the sample in the testing solution (6.3) between the electrodes. Parts of the sample which are not coated with the

test metal shall be masked before the dipping. Alternatively, immerse only the parts of the object which are coated with the test metal.

Connect the sample as anode. Carry out the electrolysis for 20 s at a current density of 1,0 mA/cm².

6.5.2 Drying

When the sample has been removed from the electrolyte, leave the gelatine coating to dry at room temperature for about 30 min; alternatively, accelerated drying may be carried out for about 15 min in an air stream at room temperature. The sample shall remain in the horizontal position.

6.5.3 Evaluation

Examine the surface for pores. Pores in the coating will appear as coloured spots, often having a strongly marked centre point of a diameter of 0,2 mm.

The colour of the spots will depend upon the substrate and/or undercoat upon which the gold or gold alloy coating has been applied, red with dark red centre spots on nickels, green with dark green centre spots on copper or copper alloys.

NOTE — When using nickel as an undercoat on copper or copper alloys, green spots with dark green points appear if the porosity extends to the basis metal.

6.6 Expression of results

Express the porosity as x pores/cm².

7 Test report

The test report shall include at least the following information :

- a) a reference to this part of ISO 4524, including an identification of the specific method used;
- b) the result(s) of the test(s) carried out and the form in which these are expressed;
- c) any unusual features noticed during the determination;
- d) any operation not included in this part of ISO 4524;
- e) any other relevant information requested by the purchaser.

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