SLOVENSKI STANDARD
SIST EN ISO 4524-3:1999

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Kovinske prevleke - Preskusne metode za galvansko nanašanje zlata in njegovih zlitin - 3. del: Elektrografsko ugotavljanje poroznosti (ISO 4524-3:1985)

Metallic coatings - Test methods for electrodeposited gold and gold alloy coatings - Part 3: Electrographic tests for porosity (ISO 4524-3:1985)

Metallische Überzüge - Prüfverfahren für elektrolytisch abgeschiedene Überzüge aus Gold und Goldlegierungen - Teil 3: Elektrographische Prüfungen auf Porosität (ISO 4524 -3:1985)

Revetements métalliques - Méthodes d'essai des dépôts électrolytiques d'or et d'alliages d'or - Partie 3: Détermination électrographique de la porosité (ISO 4524-3:1985)

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English version

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Standard has been taken over by the Technical Committee CEN/TC 262 "Protection of metallic materials against corrosion" from the work of ISO/TC 107 "Metallic and other inorganic coatings" of the International Organization for Standardization (ISO).

This document was submitted to the formal vote and was adopted by CEN as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 1995, and conflicting national standards shall be withdrawn at the latest by July 1995.

In accordance with the CEN/CENELEC Internal Regulations, following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of the International Standard ISO 4524-3:1985 has been approved by CEN as a European Standard without any modification.

International Standard 4524/3

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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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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 ($CdCl_2 \cdot 2{,}5\ H_2O$) 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/cm^2 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/cm^2 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 (Na_2CO_3) 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 ($NaNO_3$).

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.