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Metallic coatings — Review of porosity tests

Revêtements métalliques — Passage en revue des essais de 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10308 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

Annex A forms an integral part of this International Standard. Annexes B to G are for information only.

Metallic coatings — Review of porosity tests

1 Scope

This International Standard reviews published methods for revealing pores (see ISO 2080) and discontinuities in coatings of aluminium, anodized aluminium, brass, cadmium, chromium, cobalt, copper, gold, indium, lead, nickel, nickel-boron, nickel-cobalt, nickel-iron, nickel-phosphorus, palladium, platinum, vitreous or porcelain enamel, rhodium, silver, tin, tin-lead, tin-nickel, tin-zinc, zinc and chromate or phosphate conversion coatings (including associated organic films) on aluminium, beryllium-copper, brass, copper, iron, kovar (NiFeCo) alloys, magnesium, nickel, nickel-boron, nickel-phosphorus, phosphor-bronze, silver, steel, tin-nickel and zinc alloy base metal.

The tests summarized in this International Standard are designed to react with the substrate when exposed, by a discontinuity, in such a way as to form an observable reaction product.

NOTES

1 Pores are usually perpendicular to the coating surface but may be inclined to the coating surface. They are frequently cylindrical in shape but may also assume a twisted shape (see annex C).

2 Porosity may vary in size from the submicroscopic, invisible under a light microscope, to the microscopic, visible from × 10 to × 1 000, to the macroscopic, visible to the naked eye.

3 Porosity may be visibly indicated by discoloration of the coated surface.

4 Porosity in a coating is not always detrimental. In microdiscontinuous chromium, for example, porosity or microcracking is required and tests are conducted to indicate the pores.

5 Results obtained from porosity tests expressed in terms such as pores per square centimetre are relative values related to the specific test method used and the magnification used during examination. Annex B gives typical report criteria.

2 Normative references

The following standards contain provisions which, through references in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1456:1988, *Metallic coatings — Electrodeposited coatings of nickel plus chromium and of copper plus nickel plus chromium.*

ISO 2080:1981, *Electroplating and related processes — Vocabulary.*

ISO 2085:1976, *Anodizing of aluminium and its alloys — Check of continuity of thin anodic oxide coatings — Copper sulphate test.*

ISO 3160-2:1992, *Watch cases and accessories — Gold alloy coverings — Part 2: Determination of fineness, thickness, corrosion resistance and adhesion.*

ISO 4524-2:1985, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 2: Environmental tests.*

ISO 4524-3:1985, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 3: Electrographic tests for porosity.*

ISO 4525:1985, *Metallic coatings — Electroplated coatings of nickel plus chromium on plastics materials.*

ISO 4526:1984, *Metallic coatings — Electroplated coatings of nickel for engineering purposes.*

ISO 4527:1987, *Autocatalytic nickel-phosphorus coatings — Specification and test methods.*

ISO 4538:1978, *Metallic coatings — Thioacetamide corrosion test (TAA test).*

ISO 4540:1980, *Metallic coatings — Coatings cathodic to the substrate — Ratings of electroplated test specimens subjected to corrosion tests.*

ISO 4541:1978, *Metallic and other non-organic coatings — Corrodkote corrosion test (CORR test).*

ISO 6158:1984, *Metallic coatings — Electroplated coatings of chromium for engineering purposes.*

ISO 6988:1985, *Metallic and other non-organic coatings — Sulfur dioxide test with general condensation of moisture.*

ISO 9227:1990, *Corrosion tests in artificial atmospheres — Salt spray tests.*

ISO 10309:1994, *Metallic coatings — Porosity tests — Ferroxyl test.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 2080 as well as the following apply.

3.1 discontinuities: Cracks, micro-holes, pits, scratches or any other opening in the coating surface that exposes a different underlying metal.

For further information on discontinuities see annex D and reference [1] in annex G.

4 Principle

Porosity test results occur as chemical reaction end-products. Some occur *in situ*, others on paper or in a gel coating. Observations are made that are consistent with the test method and the items being tested as specified by the buyer. These may be visual inspections (naked eye) or at × 10 magnification (microscope). Other methods may involve enlarged photographs or photo-micrographs. See references [1, 2, 3, 5 and 6] in annex G (see also annex A for a tabular summary of the porosity tests and annex D for a classification of discontinuities).

5 Common features of porosity tests

Porosity tests differ from corrosion and ageing tests particularly concerning testing time. Porosity tests are primarily short-time tests. A good porosity test process must clean, depolarize and activate the substrate metal exposed by the pore and attack it to such a degree as to cause the reaction product to fill the pore to the surface of the coating. Ideally, the corrosive provided for this should not react with the coating surface. It is essential that the time of reaction be limited, particularly with thin coatings, since the corrosive will attack the substrate in all directions and in so doing, will undermine the coatings such that false observations will be made. When the corrosion product is soluble in the reagent, the precipitating indicator is used to form the reaction product. (See annex E for classification of methods of porosity testing.)

6 Test specimens

Porosity tests are generally destructive in nature and are designed to assess the quality of the coating process of the substrate. Therefore, separate test specimens are not ordinarily allowed.

7 Specific porosity tests

7.1 Alizarin test

7.1.1 Scope

For coatings of chromium (including Cr/Ni/Cu and Cr/Ni/Ni), cobalt, copper, nickel, nickel-boron, nickel-cobalt, nickel-iron and nickel-phosphorus on aluminium substrate.

7.1.2 Summary of method

The test specimens are treated with sodium hydroxide, sodium alizarin sulfonate and glacial acetic acid under defined conditions. Formation of red markings or spots indicates porosity. Details of the test procedure can be found in ISO 4527. See also references [9, 31 and 37] in annex G.

7.2 Anthraquinone test

7.2.1 Scope

For coatings of chromium (including Cr/Ni/Ni), cobalt, nickel, nickel-boron, nickel-cobalt, nickel-iron and nickel-phosphorus on aluminium, magnesium or zinc alloy substrates.

7.2.2 Summary of method

The test specimens are treated with sodium hydroxide and potassium 1-aminoanthraquinone-2-carboxylic acid under defined conditions. Formation of red markings or spots indicates porosity. Details of the test procedure can be found in reference [13] in annex G.

7.3 Cadmium sulfide test

7.3.1 Scope

For metallic coatings of chromium (including Cr/Ni/Ni), gold, palladium, platinum and rhodium on beryllium-copper, brass, copper, phosphor-bronze and silver substrates.

7.3.2 Summary of method

Filter paper is soaked in cadmium chloride and then treated with sodium sulfide to precipitate cadmium sulfide. Details of the test procedure can be found in ISO 4524-3.

7.4 Copper sulfate (Preece) test

7.4.1 Scope

Variation A. For coatings of cadmium and zinc on iron, steel or iron-based alloy substrates.

Variation B. For thin (< 5 µm) anodic oxide coatings on aluminium alloy substrates.

7.4.2 Summary of method

The test specimen is immersed in a solution of copper sulfate; different solution compositions are used for aluminium alloy and iron alloy substrates. Reddish markings or spots, of copper, indicate pores on ferrous substrates; black markings or spots indicate pores on aluminium alloy substrates. Details of the test procedures can be found in reference [38] in annex G and also in ISO 2085.

7.5 Copper sulfate (Dupernell) test

7.5.1 Scope

For coatings of chromium and micro-cracked or micro-porous chromium on nickel/copper or nickel/nickel on aluminium, iron, steel or zinc alloy substrates.

7.5.2 Summary of method

The test specimen is made the cathode in an acid copper plating bath. Copper is deposited only where the base metal or undercoat is exposed, the chromium remaining passive. Details of the test procedure can be found in ISO 1456, ISO 4525 and ISO 6158. See also references [40 and 41] in annex G.

7.6 Corrodkote test (CORR)

7.6.1 Scope

For coatings of chromium and micro-cracked or micro-porous chromium on nickel/copper or nickel/nickel on aluminium alloy, plastic, steel and iron alloy or zinc alloy substrates.

7.6.2 Summary of method

The test specimen is coated with a slurry of corrosive salts and dried. The coated specimens are then exposed to high relative humidity for a specified period of time. Details of the test procedure can be found in ISO 4541. See also references [26 and 51] in annex G.

7.7 Electrographic tests

7.7.1 Scope

Variation A. Acrylamide electrography (See warning in 7.7.2.)

For gold coatings on nickel and silver, or nickel coatings on copper substrates.

Variation B. Gel bulk electrography.

For gold, cobalt, nickel and palladium coatings on copper; gold, copper, cobalt and palladium coatings on nickel; gold on silver substrates.

Variation C. Paper electrography.

For the following combinations of indicator-coatings/substrate that have flat or nearly flat surfaces.

Indicator	Coating/substrate
1. Cadmium sulfide	Chromium, gold, palladium, platinum and rhodium on beryllium-copper, brass, copper, phosphor-bronze and silver substrates
2. Dimethylglyoxime	Gold, palladium, platinum, rhodium and silver on brass, beryllium-copper, copper, phosphor-bronze, nickel, nickel-boron and nickel-phosphorus substrates
3. Dithiooxamide	Chromium, gold, palladium, platinum and rhodium on beryllium-copper, brass, copper and phosphor-bronze substrates
4. Nioxime	Gold, palladium, platinum and rhodium on nickel, nickel-boron, nickel-iron, nickel-phosphorus and tin-nickel substrates
5. Potassium Ferrocyanide	Chromium, gold, palladium, platinum and rhodium on brass, beryllium-copper, copper and phosphor-bronze substrates
6. Potassium Ferricyanide	Cadmium, nickel, tin and zinc on brass, silver and steel substrates

7. Magneson	Chromium, cobalt, copper, nickel, nickel-boron, nickel-cobalt, nickel-iron and nickel-phosphorus on magnesium substrates

7.7.2 Summary of methods

Variation A. Acrylamide electrography.

Acrylamide solution containing a hardener and an indicator is poured on to the sample shortly before gelatinizing. The sample is made the anode in a cell with a chloride solution and electrolized. Pores are revealed as coloured marks or spots. Details of the test procedure can be found in reference [7] in annex G.

WARNING — Acrylamide has been identified as a neurotoxin and carcinogen; use with extreme caution.

Variation B. Gel bulk electrography.

A mixture of clear gelatine, conducting salts and an indicator are poured into an electrolytic cell with a gold or platinum cathode and with the specimen as the anode. The composite gel solution is allowed to solidify following which the cell is electrolized. Pores are revealed as coloured marks or spots. Details of the test procedure can be found in reference [52] in annex G.

Variation C. Paper electrography.

Test specimens are sandwiched as an anode between electrolyte-soaked paper and indicator paper and clamped with two cathode covers (of non-reactive materials such as gold or stainless steel). A specified current (usually 0,15 milliamps/cm^2 to 1,55 milliamps/cm^2) for a specified time (usually 10 s to 30 s) is applied. After exposure, the test paper is wetted with indicator and allowed to dry.

A variety of commercially prepared test papers is available. Details of the test procedures can be found in ISO 4524-3. See also references [15, 18, 35, 36 and 42] in annex G.

7.8 Ferrocyanide test

7.8.1 Scope

For coatings of chromium, cobalt, gold, nickel, nickel-boron, nickel-iron, nickel-phosphorus, palladium, platinum and rhodium on copper substrate.

7.8.2 Summary of method

The test specimens are treated with glacial acetic acid and potassium ferrocyanide under defined conditions. Formation of brown markings or spots indicates porosity. Details of the test procedure can be found in ISO 4527. See also references [12 and 37] in annex G.

7.9 Ferron test

7.9.1 Scope

For coatings of aluminium, brass, cadmium, chromium, cobalt, indium, lead, nickel, nickel-boron, nickel-phosphorus, porcelain enamel, organic films, silver, tin, tin-lead, tin-nickel, tin-zinc and zinc, on iron and steel substrates.

7.9.2 Summary of method

The test specimens are treated with acid and a 0,1 % solution of ferron (8-hydroxyquinoline 7-iodo-5-sulfonic acid), under defined conditions. Formation of red markings or spots indicates porosity. Details of the test procedure can be found in reference [4] in annex G.

7.10 Ferroxyl test

7.10.1 Scope

For metallic coatings such as brass, chromium, cobalt, copper, gold, indium, lead, nickel, nickel-boron, nickel-phosphorus, organic films, porcelain enamel, silver, tin, tin-lead and tin-nickel that are resistant, for the duration of the test period, to ferricyanide and chloride and are also cathodic to their iron or steel alloy substrates.

7.10.2 Summary of method

Electrolyte-wetted, gel-chloride-treated paper strips are placed firmly in contact with test specimen surfaces for a specified time. After the allotted time, the paper strips are wetted adequately with ferricyanide indicator solution. Blue markings or spots indicate pores. Details of the test procedure can be found in ISO 4526, ISO 4527 and ISO 10309. See also references [29, 35, 36, 39 and 43] in annex G.

7.11 Flowers of sulfur porosity test

7.11.1 Scope

Primarily for gold and nickel coatings on copper, copper alloys or silver substrates. It may be used with other coatings that do not significantly tarnish in reduced sulfur atmospheres.

7.11.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive container with controlled humidity and elevated temperature (50 °C) for a specified time in a closed system over the flowers of sulfur. Markings, spots or discoloration indicate

porosity. Details of the test procedure can be found in reference [44] in annex G.

7.12 Hot water test

7.12.1 Scope

For metallic coatings cathodic to a ferrous substrate; for example, brass, copper, gold, indium, nickel, nickel-boron, nickel-phosphorus, tin, tin-lead and tin-nickel on iron, kovar (NiFeCo) alloys or steel substrates; vitreous or porcelain enamel and organic films on steel substrates.

7.12.2 Summary of method

The test specimens are placed in a glass vessel filled with neutral, aerated water at room temperature. Heat is applied to the vessel at a rate that will bring the water to a boil in 15 min to 20 min. Boiling is continued for 30 min. After exposure and drying, black markings or spots and red rust indicate porosity. Details of the test procedure can be found in ISO 4527. See also references [37 and 43] in annex G.

7.13 Hydrogen sulfide or sulfur dioxide/hydrogen sulfide test

7.13.1 Scope

Variation A. For coatings of less than 5 µm of gold, palladium or rhodium on beryllium-copper, brass, copper, phosphor-bronze and silver substrates.

Variation B. For coatings of more than 5 µm of gold, palladium, rhodium, tin, tin-lead, or tin-nickel on beryllium-copper, brass, copper, nickel, nickel-boron, nickel-phosphorus, phosphor-bronze or silver substrates.

7.13.2 Summary of method

Variation A. Test specimens are suspended on non-reactive supports in a non-reactive container with a freshly generated hydrogen sulfide atmosphere for a specified time, usually 24 h. Discoloration on the surface indicates porosity. Details of the test procedure can be found in reference [27] in annex G.

Variation B. Test specimens are suspended on non-reactive supports in a non-reactive container with a freshly generated sulfur dioxide atmosphere for a specified time, usually 24 h. Followed by freshly generated hydrogen sulfide atmosphere for a specified time, usually 24 h. Discoloration on the surface indicates porosity. Details of the test procedure can be found in reference [17] in annex G.

7.14 Logwood test

7.14.1 Scope

For coatings of brass on aluminium; or silver on brass and copper substrates.

7.14.2 Summary of method

Paper strips treated with haematoxylin are immersed in water and are placed firmly in contact with test specimen surfaces for a specified time. After the allotted time, the paper strips are examined for blue markings or spots which indicate pores. Details of this test can be found in references [8 and 11] in annex G.

7.15 Magneson test

7.15.1 Scope

For coatings of chromium, cobalt, copper, nickel, nickel-boron, nickel-cobalt, nickel-iron and nickel-phosphorus on magnesium substrates.

7.15.2 Summary of method

The test specimens are treated with sodium hydroxide. Dry magneson test paper, prepared by dipping filter paper in a 0,01 % alcohol solution of *p*-nitrobenzene-azo-resorcinol is applied to the treated surface. Formation of blue markings or spots on a red background indicates porosity. Details of the test procedure can be found in reference [15] in annex G.

7.16 Nitric acid vapour test

7.16.1 Scope

For gold coatings on beryllium-copper, brass, copper, nickel, nickel-boron, nickel-phosphorus, phosphor-bronze and tin-nickel substrates.

7.16.2 Summary of method

A stabilized acid atmosphere is established by placing concentrated nitric acid in a non-reactive container, which is covered and left to stand for 0,5 h at a specified ambience.

Test specimens are suspended in this closed system atmosphere and exposed for a specified exposure time; 1 h for copper alloy substrates and 2 h for nickel-based substrates. After exposure the specimens are baked to dry and fix the reaction products. Each reaction product marking or spot, usually protruding, indicates a pore in the coating. Details of the test procedure can be found in ISO 4524-2. See also reference [45] in annex G.

7.17 Oxine test

7.17.1 Scope

For coatings of chromium, cobalt, copper, nickel, nickel-boron, nickel-cobalt, nickel-iron and nickel-phosphorus on aluminium, magnesium and zinc substrates.

7.17.2 Summary of method

The test specimens are treated with sodium hydroxide. Dry oxine test paper, prepared by dipping filter paper in a 5 % alcohol solution of 8-hydroxyquinoline is applied to the treated surface. Formation of coloured markings or spots indicates porosity. Details of the test procedure can be found in references [10 and 14] in annex G.

7.18 Permanganate test

7.18.1 Scope

For coatings of aluminium, cadmium and zinc on iron, steel or iron-based alloy substrates.

7.18.2 Summary of method

The test specimen is immersed in a dilute solution of potassium permanganate. Black markings or spots, of manganese dioxide, indicate pores. Details of this test can be found in reference [8] in annex G.

7.19 Polysulfide test

7.19.1 Scope

For metallic coatings of tin, tin-nickel and tin-zinc on beryllium-copper , brass, copper and phosphor-bronze substrates.

7.19.2 Summary of method

Coated parts are solvent-cleaned and then immersed in a solution of sodium polysulfide. Formation of black marks or spots indicates pores. Details of the test procedure can be found in reference [46] in annex G.

7.20 Porotest test

7.20.1 Scope

For metallic coatings such as brass, chromium, copper, gold, nickel, nickel-boron, nickel-phosphorus, tin, tin-nickel and their alloys which are cathodic to their iron, steel or iron-based alloy substrates.

7.20.2 Summary of method

Paper strips, treated with α-nitroso-β-naphthol, are immersed in water — or, to speed up the reaction, 5 % sodium chloride — are placed firmly in contact with test specimen surfaces for a specified time. After the allotted time, the paper strips are examined for green markings or spots which indicate pores. Details of this test can be found in reference [8] in annex G.

7.21 Salt spray tests [neutral (NSS), acetic (AASS) and cuproacetic (CASS)]

7.21.1 Scope

For metallic coatings such as brass, chromium, cobalt, copper, gold, lead, nickel, nickel-boron, nickel-phosphorus, tin, tin-lead and tin-nickel that are resistant, for the duration of the test period, to chloride and which are also cathodic to their iron, steel, or iron-based alloy substrates. The test is also suitable for coatings of chromium on nickel/copper and chromium on nickel/nickel on aluminium, magnesium and zinc substrates.

7.21.2 Summary of method

Specimens are placed in a cabinet and subjected to a fog spray of 5 % sodium chloride solutions. Porosity is indicated by black markings or spots and red rust on substrates of iron, steel or iron-based alloys, or white markings on spots, or blisters in the coating on substrates of aluminium, magnesium or zinc alloy (see ISO 4540). Details of the test procedure can be found in ISO 9227. See also references [32, 33, 47 and 48] in annex G.

7.22 Sulfur dioxide test

7.22.1 Scope

Variation A. For coatings of gold on copper, copper alloys and nickel substrates.

Variation B. For coatings of gold on silver substrates.

Variation C. For coatings of tin, tin-lead and tin-nickel on copper, copper alloy and steel substrates.

7.22.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive container with a freshly generated sulfur dioxide atmosphere for a specified time, usually 24 h. The concentrations of sulfur dioxide, generated as the corrosive atmosphere, are selected for the specific variation A, B or C (coating and substrate combination). Colour on the surface indicates porosity. Details of the test procedure can be found in ISO 4524-2 and ISO 6988. See also references [28, 32, 35, 49 and 50] in annex G.

7.23 Sulfurous acid/sulfur dioxide vapour test

7.23.1 Scope

For coatings of gold and palladium on beryllium-copper, brass, copper, nickel, nickel-boron, nickel-phosphorus and phosphor-bronze substrates.

7.23.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive container with a sulfurous acid/sulfur dioxide atmosphere for a specified time, usually 24 h. Colour on the surface indicates porosity. Details of the test procedure can be found in reference [53] in annex G.

7.24 Thiocyanate test

7.24.1 Scope

For metallic coatings such as chromium, copper, nickel, nickel-boron, nickel-phosphorus, tin, tin-nickel and their alloys that are resistant to thiocyanate and chloride during the time period of the test and which are also cathodic to their iron or steel alloy substrates.

7.24.2 Summary of method

Electrolyte-wetted, gel-chloride-treated paper strips are placed firmly in contact with test specimen surfaces for a specified time. After the allotted time, the paper strips are wetted adequately with thiocyanate indicator solution. Red markings or spots indicate pores. Details of this test procedure can be found in reference [8] in annex G.

7.25 Thioacetamide test (TAA)

7.25.1 Scope

For gold, nickel or tin coatings on copper, copper alloys and silver substrates. This test can also be used for organic coatings on brass, copper or silver substrates.

7.25.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive container with a saturated solution of sodium acetate, for controlling humidity (75 % RH), and crystals of thioacetamide at 25 °C for a specified time in the closed system. Markings, spots or discoloration indicate porosity. Details of the test procedure can be found in ISO 4538. See also reference [16] in annex G.

7.26 Watch case acetic acid test

7.26.1 Scope

For gold coatings on cuprous alloy with or without nickel, and die-cast zinc-based alloy substrates.

7.26.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive vessel and exposed to vapours of acetic acid for 24 h at 23 °C ± 2 °C. Details of the test procedure can be found in ISO 3160-2.

7.27 Watch case sodium bisulfite test

7.27.1 Scope

For gold coatings on ferrous alloy substrates.

7.27.2 Summary of method

Test specimens are suspended on non-reactive supports in a non-reactive vessel and exposed to vapours of sodium bisulfite for 24 h at 23 °C ± 2 °C. Details of the test procedure can be found in ISO 3160-2.