INTERNATIONAL STANDARD

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 10308:1995), which has been technically revised.

Metallic coatings — Review of porosity tests

WARNING — This international Standard calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves either the designer, the producer, the supplier or the user from statutory and all other legal obligations relating to health and safety at any stage of manufacture or use.

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, NiFeCo alloys, magnesium, nickel, nickel-boron, nickel-phosphorus, phosphor-bronze, silver, steel, tin-nickel and zinc alloy basis 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.

NOTE 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).

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

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

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

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 10289:1999, *Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2080 and 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

NOTE For further information on discontinuities, see Annex D and reference [1] in the Bibliography.

4 Principle

The results of porosity tests are the end products of a chemical reaction with a metallic substrate. 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 purchaser. 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 the Bibliography (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 tests and, particularly, ageing tests regarding test duration. Porosity tests are primarily short-time tests. A good porosity test process shall 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. The corrosive shall not react with the coating. 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 resulting in misleading observations. When the corrosion product is soluble in the reagent, the precipitating indicator is used to form the reaction product. (See Annex E for a 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, as a rule, separate test specimens are not used.

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 references [9, 21 and 25] in the Bibliography.

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. The formation of red markings or spots indicates porosity. Details of the test procedure can be found in reference [13] in the Bibliography.

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. The sample is sandwiched between the cadmium sulfide paper (which acts as the anode) and the moistened blotting paper fastened to a high-purity clean aluminium or stainless steel platen (which acts as the cathode). D.C. current is applied for a specific time. Brown stains on the paper indicate pores. 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 and 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 ISO 2085. See also reference [26] in the Bibliography.

7.5 Copper sulfate (Dupernell) test

7.5.1 Scope

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

7.5.2 Summary of method

The test specimen is used as the cathode in an acid copper plating bath. Copper is deposited only where the basis metal or the substrate is exposed, the chromium remaining passive. After the test, examine the surface for cracks using an optical microscope. Details of the test procedure can be found in ISO 1456, ISO 4525 and ISO 6158. See also references [27 and 28] in the Bibliography.

7.6 Corrodkote test (CORR)

7.6.1 Scope

For coatings of chromium and micro-cracked or microporous 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 exposed to high relative humidity for a specified period of time, then cleaned and treated for redeveloping the points of failure, e.g. in a salt spray cabinet. Porosity is indicated by black markings or red rust on the iron-based substrates or by white markings on the aluminium and zinc substrates (see ISO 10289). Details of the test procedure can be found in ISO 4541. See also reference [38] in the Bibliography.

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. Dithioxamide	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 onto 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 the Bibliography.

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

Variation B. Gel bulk electrography.

A mixture of clear gelatin, 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 spots or blooms. Details of the test procedure can be found in ISO 15720. See also reference [39] in the Bibliography.

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 mA/cm^2 to 1,55 mA/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. Pores are revealed as coloured spots.

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, 24 and 29] in the Bibliography.

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 references [12 and 37] in the Bibliography.

7.9 Ferron test

7.9.1 Scope

For coatings of aluminium, brass, cadmium, chromium, cobalt, indium, lead, nickel, nickel-boron, nickel-phosphorus, 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 the Bibliography.

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, 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 and ISO 10309. See also references [20 and 30] in the Bibliography.

7.11 Flowers-of-sulfur porosity test

7.11.1 Scope

For coatings of gold, nickel, tin, tin-lead, palladium and their alloys on copper, copper alloy or silver substrates. It may be used with other coatings that do not significantly tarnish in a reduced-sulfur atmosphere.

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. Brown or black tarnish marks or spots indicate porosity. Details of the test procedure can be found in ISO 12687. See also reference [31] in the Bibliography.

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, NiFeCo alloys or steel substrates; organic films on steel substrate.

7.12.2 Summary of method

The test specimens are placed in a glass vessel filled with distilled or deionized and aerated water (pH 6,0 to 7,5, conductivity not higher than 0,5 mS/m). Water is heated to 85 °C and the temperature shall be maintained throughout 60 min (test period). After exposure and drying, black markings or spots and red rust indicate porosity. Details of the test procedure can be found in ISO 4526. See also references [25 and 30] in the Bibliography.

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. Discolouration on the surface indicates porosity. Details of the test procedure can be found in reference [41] in the Bibliography. See also reference [53] in the Bibliography.

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. Discolouration on the surface indicates porosity. Details of the test procedure can be found in reference [17] in the Bibliography.

7.14 Haematoxylin 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 the Bibliography.

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 substrate.

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 ρ-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 the Bibliography.

7.16 Nitric acid vapour test

7.16.1 Scope

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

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 60 min $\pm$ 5 min. After exposure, the specimens are heated to dry 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 14647. See also reference [32] in the Bibliography.

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 the Bibliography.

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 the Bibliography.

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 markings or spots indicates pores. Details of the test procedure can be found in reference [32] in the Bibliography.

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 the Bibliography.