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Metallic coatings — Porosity tests — Porosity in gold or palladium coatings on metal substrates by gel-bulk electrography

Revêtements métalliques — Essais de porosité — Porosité des revêtements d'or ou de palladium sur métaux par électrographie par gélification

Reference number
ISO 15720:2001(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15720 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

Annexes A and B of this International Standard are for information only.

Introduction

This test method is an electrographic technique, "gel-bulk electrography." The specimen is made the anode in a cell containing a solid or semi-solid electrolyte of gelatin, conducting salts and an indicator. Application of current to this cell results in the migration of base metal ions through continuous pores. Reaction of cations with an indicator gives rise to coloured *reaction products* (not to be confused with *corrosion products)* at pore sites which may be counted through the clear gel. Individual spots are counted with the aid of a lense or low-power stereomicroscope.

The test method is designed to show whether the porosity level is less than or greater than a given value which, from experience, is considered by the user to be acceptable for the intended application.

These porosity tests involve corrosion reactions in which the products delineate defect sites in coatings. Since the chemistry and properties of these products do not resemble those found in natural or service environments, these tests can not be recommended for the prediction of the electrical performance of contacts unless correlation is first established with service experience.

This test method is suitable for coatings containing 75 % or more of gold on substrates of silver, nickel, copper and its alloys that are commonly used in electrical contacts. This test method is also suitable for coatings of 95 % or more of palladium on nickel, copper and its alloys, and for coatings of palladium-nickel alloy (75 % or more of palladium) on nickel, copper and its alloys.

This test method is capable of detecting porosity or other defects in gold or palladium coatings that could participate in substrate corrosion reactions. In addition, it can be used on contacts having complex geometry such as pin-socket contacts (although difficulty may be experienced in inspecting deep recesses).

This test is considered destructive because it reveals the presence of porosity by contaminating the surface with corrosion products and by it undercuts the corrodible metal at pore sites and at unplated areas. In addition, the surface is coated with a corrosive gel mixture which is difficult to remove completely. Any parts exposed to the gel test shall not be placed in service.

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Metallic coatings — Porosity tests — Porosity in gold or palladium coatings on metal substrates by gel-bulk electrography

WARNING — This International Standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see clause 6.

1 Scope

This test method covers equipment and techniques for determining porosity in noble metal coatings, particularly electrodeposits of gold, palladium and palladium-nickel alloy (70 % to 90 % palladium) and clad metals used on electrical contacts.

The gel-bulk procedure is not as sensitive to small pores and is more complex than porosity tests involving gaseous corrodants (see ISO 14647 and ISO 15721). It also involves more chemicals, preparation and auxiliary equipment.

This test is intended to be used for quantitative descriptions of porosity (such as number of pores per unit area or per contact) only on measurement areas where coatings have a sufficiently low pore density such that the corrosion sites are well separated and can be readily resolved. As a general guideline this can be achieved for pore densities up to about 25/cm^2.

Other porosity testing methods are outlined in ISO 10308. Detailed critical reviews of porosity testing are also available. Other porosity test methods are described in ISO 12687, ISO 14647 and ISO 15721.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness.*

ISO 2079, *Surface treatment and metallic coatings — General classification of terms*.

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

ISO 10308, *Metallic coatings — Review of porosity tests*.

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 2064, ISO 2079, ISO 2080 and ISO 10308 as well as the following apply.

3.1
decorations
coloured reaction products emanating from the pores that provide visual contrast with the gel medium

NOTE While non-standard terms are deprecated, the term decorations has had long-standing use in differentiating between coloured pore indicators and true corrosion products. The term can be found in the technical literature.

3.2
metallic coatings
Includes platings, claddings, or other metallic layers applied to the substrate

NOTE The coatings can comprise a single metallic layer or a combination of metallic layers.

3.3
porosity
The presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal

4 Apparatus

4.1 Test vessel, of glass, acrylic resin or other inert colourless transparent material. It shall have thin-walled flat sides and be of a size appropriate to the sample to be tested.

4.2 Power supply, rated at 0 A to 1 A and 0 V to 10 V dc; an electronically regulated, constant-current (± 5 %) apparatus is preferred.

4.3 dc milliammeter.

4.4 Separate dc voltmeter.

4.5 Cathode material, in the form of plain expanded foil or wire and made of titanium and coated with platinum, gold or other inert coating. Alternatively, platinum, gold or other inert metals may be used.

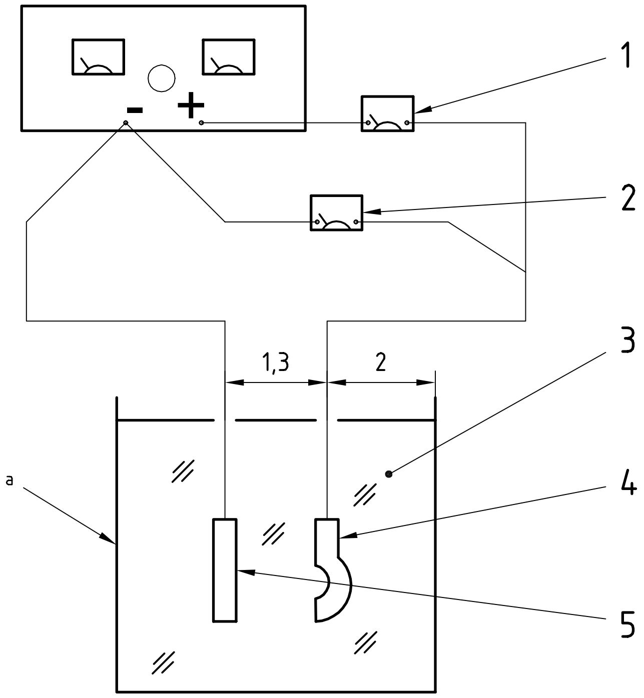
The cathode and specimen (anode) areas shall be approximately the same. Additional, gold or platinum wires for cathode and anode are needed for that portion of the electrical connection that is in the reagent solution. If small alligator clips are used to secure the lead wires to the cathode and anode they shall be heavily gold-plated so as to be entirely free of porosity. A variation of this procedure, suitable for samples with relatively few pores, is to use a second identical test sample as the cathode. The test can be run with current first in the forward, then in the reverse direction so that the porosity in both samples may be determined. Figure 1 is a diagram of the test cell setup.

NOTE A commonly used alternative cell design incorporates the cathode as part of the cell structure (as shown in Figure 2). In addition, the samples may be attached to a common carrier strip or holder, so that only the sample surfaces need be in the gel.

4.6 Timer, capable of indicating seconds.

4.7 Stereomicroscope, with ×10 magnification and an illuminator for sample inspection after test. An eyepiece reticule is recommended for convenience in locating the contact area or other significant measurement areas.

Dimensions in millimetres

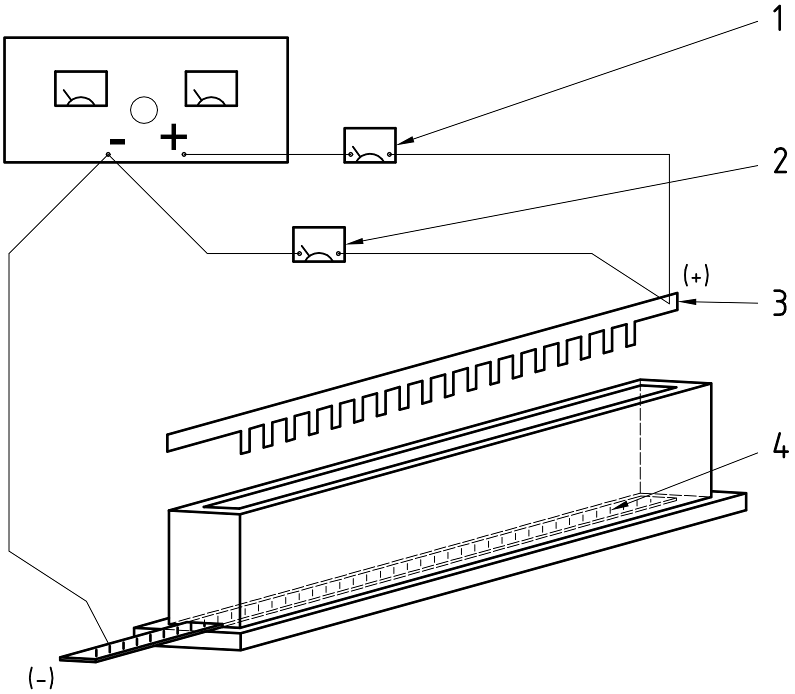


Key

1 dc milliammeter (4.3)
2 dc voltmeter (4.4)
3 Gel
4 Contacts (anode)
5 Cathode (approximately same surface area as specimens under test)

a Observation at ×10 magnification of components *in situ*.

Figure 1 — Schematic representation of typical test-cell set-up; anode (sample) and cathode are facing each other (preferred orientation)



Key

1 dc milliammeter (4.3)
2 dc voltmeter (4.4)
3 Sample strip (anode)
4 Cathode

Figure 2 — Exploded view of alternate design with cathode incorporated as part of cell structure

5 Reagent

5.1 General

Some of the indicating reagents (see Table 1) are sensitive to heat and light, particularly rubeanic acid (dithiooxamide). The indicator solutions shall be stored in dark stoppered bottles. For rubeanic acid, it shall not be stored longer than one month and shall be filtered prior to use.

5.2 Food-grade gelatin

This type is preferred to chemically pure grade gelatin because the latter may not give transparent solutions. A 10 % solution is prepared by mixing 9 g of the gelatin in 91 ml of distilled or deionized water and slowly heating to 60 °C to 65 °C while stirring, until all the gelatin dissolves.

NOTE If the storage bottle is tightly capped, the plain gelatin solution may be stored for up to 2 d in a refrigerator and kept at 5 °C to 10 °C, discard it if mold appears on its surface.

6 Safety hazards

Reagents listed in Table 1 have the potential to cause injury or skin discoloring if improperly handled. Good laboratory practice including the use of a fume cupboard and skin and eye protection shall be observed, especially during preparation of the solution and the cleaning of the test samples. Proper precautions in the use of electrical power supplies and electrical connections shall also be scrupulously observed.

Table 1 — Guide to gel porosity testing solutions

Test for	Test solution	Indicator	Indicating colour	Remarks
Copper A	4 % sodium carbonate + 1 % sodium nitrate	saturated solution of rubeanic acid in ethanol	dark olive green	also detects nickel, cobalt
Copper	4 % sodium carbonate + 1 % sodium nitrate	7,5 % potassium ferrocyanide in water	brown	
Nickel A	4 % sodium carbonate + 1 % sodium nitrate	saturated solution of rubeanic acid in ethanol	blue/blue-violet	also detects copper, cobalt
Nickel	5 % ammonium hydroxide	saturated solution of dimethylglyoxime in ethanol	pink	
Silver [a,b]	0,2 M nitric acid	1 % glacial acetic + 5 % sodium dichromate	red	solution must be free of halogens
Silver [a]	0,2 M nitric acid	saturated solution of rhodamine in ethanol	red/red-violet	solution must be free of halogens

[a] Preferred test.

[b] Not suitable for palladium overplates.

7 Procedure

7.1 General

This test is suitable for gold coating on silver, nickel, or copper and its alloys and palladium coat on nickel, copper and its alloys either as underlayers or substrates, in accordance with the reagents chosen from Table 1.

7.2 Sequence of operations

a) Prepare the test solution.

b) Prepare the indicator.

c) Calculate the current to be used.

d) Inspect the samples prior to cleaning.

e) Clean the samples

f) Prepare the gel while the samples are cleaning. Remove from heat when dissolved.

g) Dry the samples.

h) Suspend the samples in the test cells.

i) Prepare the composite gel solution and add to the cells.

j) Solidify the gel thoroughly.

k) Set up the equipment and make electrical attachments:

  1) apply the calculated current;

  2) examine immediately for gross defects;

  3) take readings after the prescribed time period;

  4) record the results.

7.3 Cleaning

Handle specimens as little as possible and only with tweezers, microscope-lens tissue or clean, soft cotton gloves. Prior to the test, inspect the samples under 10× magnification for evidence of particulate matter. If present, such particles shall be removed by blowing them off with clean, oil-free air. If this is not successful, discard the sample and draw a replacement sample from the batch. Then, clean the samples with solvents or solutions that do not contain chlorinated hydrocarbons, CFCs or other known ozone-destroying compounds. The procedure outlined in the note in 5.2 has been found to give satisfactory results for platings with mild to moderate surface contamination.

NOTE Suggested cleaning procedure.

a) Keep individual contacts separated if there is a possibility of damaging the measurement areas during the various cleaning steps.

b) Clean samples for 5 min in an ultrasonic cleaner which contains a hot (65 °C to 85 °C) 2 % aqueous solution of a mildly alkaline (pH 7,5 to pH 10) detergent.

c) After ultrasonic cleaning, rinse samples under warm running tap water for at least 5 s.

d) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.

e) Immerse in fresh methanol or isopropanol and ultrasonically "agitate" for at least 30 s in order to remove the water from the samples.

f) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used for drying, the air shall be oil-free, clean and dry.

g) Do not touch measurement area of the samples with bare fingers after cleaning.

h) Re-inspect samples (under ×10 magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning steps. Surface cleanliness is extremely important; contaminants, such as plating salts, organic films and metal flakes, may give erroneous indications of defects and are unacceptable.

7.4 Electrographic method

Secure the specimens to be tested to gold or platinum lead wires and suspend in the cell so that their measurement areas (see B.3) can be easily seen through the wall of the test vessel (4.1). Place the specimens 13 mm ± 2 mm from the cathode.

NOTE 1 For longitudinal-shaped contacts, such as pins or posts, where the alternative cell design is used (see Figure 2) the bottom ends or tips of the contacts shall be at a distance from the cathode that is at least 1,5 times their maximum length in the gel solution. The area of the cathode along the bottom of the cell should be not less than the total specimen area in the solution and can be up to twice as large. If the cathode area is too small there will be a risk of excessive hydrogen bubble formation.

The composite test gel solution is prepared from the gel solution (see 5.2) and an indicator and test solution chosen from Table 1. The composite contains by volume, 94,6 % gel solution, 3,4 % test solution and 2 % indicator. It is prepared by melting the gelatin solution at 59 °C ± 1 °C whilst gently stirring, removing from the heat and adding, while stirring, the premixed test solution and then the indicator. Stirring shall be continued for 2 min to 3 min in order to thoroughly mix all the components. For consistency, consider the lifespan of the composite gel solution to be 2 h. Testing of the samples shall be accomplished within this time period.

Pour the composite test gel solution into the test vessel, taking care not to introduce bubbles of air into the solution. Allow the gel to solidify.

NOTE 2 It is good practice to include with the sample a control specimen that is known to be porous (such as would be produced by scratching the coating to expose substrate). If the control specimen fails to produce a dense opaque pore indication the test results must be discounted and the test repeated using fresh prepared reagents.

Attach the specimen lead to the positive terminal of the power supply and the cathode lead to its negative terminal, as shown in Figure 1 or 2.

Pass the current for 20 s using the timer (4.6) at a current density of 3,87 mA/cm^2. Current density tolerance is ± 25 %. The test shall be conducted at 23 °C ± 3 °C. During passage of the current, base metal ions are produced at pores in the test sample. These ions react with the indicator to produce coloured products in the pore sites.

NOTE 3 If bubbling of the gel should occur (highly unlikely at the low current density specified), repeat the test with new samples at a 20 % to 25 % lower current density.

NOTE 4 When calculating the current, the total metal area which is immersed in the gel should be the sample area and not the significant surfaces.

Monitor and record the maximum voltage with the voltmeter (4.4) during the application of current. The voltage shall be between 2,0 V and 2,1 V. If the voltage exceeds 3 V, the test shall be considered invalid.

8 Examination

8.1 Do not remove samples from the test vessel. Examine the measurement areas of the samples through the vessel walls (at ×10 magnification with suitable illumination) immediately after cessation of current. Note the gross defects during the first 2 min in order to determine the general location of the largest pore sites.

8.2 Begin actual pore count after 2 min and complete it within 3 min of cessation of current.

8.3 Pore sites show up as coloured dots or blooms. The size of these decorations depends on the total amount of current passed as well as the size of the defect. Bleeding or diffusion of the colour is usually slight during the first 20 min and discrete pore sites will be observed if not very close to each other. Cracks show up as continuous lines.

NOTE Annex A gives a number of guidelines that have proved useful in examining pore indications from tests that use the rubeanic acid indicator and the sodium carbonate/sodium nitrate electrolyte (Table 1).

8.4 An alternative enumeration method is to record the number of contacts with pores (in a sample strip with ten or more contacts), as defined in 8.3.

9 Precision

The precision of this procedure for five test runs in one laboratory, for palladium platings 2,2 µm - 2,4 µm thick, was 46 % (as a coefficient of variation where the total number of pores per run had a mean value of 2,8) and was even higher in subsequent determinations in other laboratories.

10 Test report

The test report shall contain the following information:

a) reference to this International Standard, i.e. ISO 15720;

b) the cleaning procedure used;

c) the nature of the coating;

d) the nature of the substrate and any intermediate underlayers;

e) the current density used;

f) the maximum voltage used;

g) the total time of current application;

h) the test solution used;

i) the indicator used;

j) the results obtained, i.e. the number, size and colour of decorations observed;

k) the results required by the relevant product standard.

Annex A
(informative)

Useful guidelines for examining samples

The following guidelines are useful when examining samples (for pore indications) with tests that use the rubeanic acid indicator and sodium carbonate/sodium nitrate electrolyte (of Table 1).

a) The reaction products give coloured blooms whose major growth takes place during the first 3 min after cessation of current. Blooms from the edges of narrow samples (e.g., posts) may obscure the measurement area, so the immediate cursory examination is important to determine pore sites. A typical site usually shows a dark spot at the centre of a coloured cloud.

b) The indicator will react with nickel and copper ions, producing coloured blooms. Copper often shows an olive or grey-green reaction product while nickel often produces a blue or blue-violet colouration.

c) When a nickel underplate is used over a copper alloy substrate, two colours may be present. Nickel ions diffuse farther (i.e. away from the pore) and may form a blue to violet haze around the central olive green or black central copper pore indication.

d) Under certain conditions, palladium platings may produce bright red-orange reaction products. These blooms should not be counted as pores unless evidence of nickel or copper pore indications are also present.

e) Very small pores and embedded particles show up as small coloured points or dots at ×10 and should be recorded separately as such.

f) Cracks show up as continuous lines and shall be recorded separately as such.

g) A hazy black cloud covering the surface without evidence of dark spots (pore sites) indicates gross defects or microcracking and should be counted as a multiple pore site.

h) When plated electrical contacts are tested together as strips or combs of contacts, the size of a pore indication is most conveniently noted as a fraction of the width of the contact surface.

Annex B
(informative)

Significance and use

Noble metal coatings, particularly gold or palladium, are often specified for the contacts of separable electrical connectors and other devices. Electrodeposits are the form of gold or palladium which is most used on contacts, although gold and palladium are also used as clad metal and as weldments on the contact surface. The intrinsic nobility of gold and, to a certain extent palladium, enables them to resist the formation of insulating films that could interfere with reliable contact operation.

In order that the nobility of gold be assured, porosity, cracks and other defects in the coating that expose substrates and underplates must be minimal or absent, except in those cases where it is feasible to use the contacts in structures that shield the surface from the environment or where corrosion inhibiting surface treatments for the deposit are used. The tolerable level of porosity in the coating depends on the severity of the environment to the underplate or substrate, design factors for the contact device like the force with which it is mated, circuit parameters, and the reliability of contact operation. When present, the location of pores on the surface is also important. If the pores are few in number or are outside the zone of contact of the mating surfaces, their presence can often be tolerated.

Methods for determining porosity on a contact surface are most suitable if they enable their precise location and numbers of pores to be determined. Contact surfaces are often curved or irregular in shape and testing methods need to be suitable for application to such curved or irregular surfaces. In addition, the severity of porosity-determining tests may vary from procedures capable of detecting all porosity to procedures that detect only gross defects. The test method in this document is generally regarded as severe.

The relationship of porosity levels to contact behaviour, revealed by particular tests, must be made by the user of these tests through practical experience or judgement. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores in the contact zone may be acceptable for others.

Bibliography

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