INTERNATIONAL
STANDARD

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Metallic coatings — Porosity tests — Ferroxyl test

Revêtements métalliques — Essais de porosité — Essai au ferroxyle

Reference number
ISO 10309:1994(E)

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 10309 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

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Metallic coatings — Porosity tests — Ferroxyl test

1 Scope

This International Standard specifies a method of revealing pores or other discontinuities, when testing metallic coatings, that are not visibly affected by ferricyanide and chloride ions during the test period and that are cathodic to iron and steel. This method is especially useful for thick, hard chromium coatings used for wear resistance.

NOTE 1 With some coating materials a very thin layer is dissolved by the sodium chloride solution during a 10 minute application period (see 5.2.3). The impact of such dissolution is that potential porosity, i.e. pores that have been covered over by very thin layers, are sometimes re-exposed. Experience has shown that such potential porosity is frequently re-exposed during actual service.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Principle

Base metal ions, formed in corrosion cells at the bottom of discontinuities in metallic coatings migrate to treated test paper held in contact with the coating surface. The base metal ions retained on the treated test paper form a blue-coloured marking or spot when the treated paper is subsequently immersed in a solution of a ferricyanide indicator solution.

4 Reagents and materials

4.1 Purity

All chemicals used shall be of a recognized analytical reagent grade and the water used shall be distilled or deionized having a conductivity not greater than 20 µS/cm (see ISO 3696).

4.2 Preparation of the indicator solution

4.2.1 Sodium chloride reagent

Dissolve 50 g of sodium chloride and 1 g of a non-ionic wetting agent in 1 litre of hot (90 °C) water. Dissolve 50 g of gelatin or agar in the above mentioned hot sodium chloride solution to provide gelling properties. The solution will then gel upon cooling, but can be re-liquefied, for use, by heating it to 35 °C.

NOTE 2 A variety of non-ionic wetting agents is commercially available.

4.2.2 Ferricyanide reagent

Dissolve 10 g of potassium hexacyanoferrate(III) (potassium ferricyanide) in 1 litre of water. Measure the pH of the solution. If it is outside the range 6 ± 0,2 discard the solution and the reagent and obtain a purer grade of reagent.

4.3 Optional equipment

A flat, flexible plastic sheet (template) with a square opening, at least 10 mm × 10 mm in size, may be used to define the test area of the test paper after the appearance of the blue coloration (development).

5 Procedure

5.1 Specimen preparation

Clean and degrease the surface area to be tested using a suitable solvent, such as 1,1,1 trichloroethane.

5.2 Treated test paper

5.2.1 Iron contamination

The paper used to prepare the test strips must be free of any iron contamination. This may be verified by first immersing the paper in sodium chloride solution, blotting, and then immersing the paper in the potassium hexacyanoferrate(III) solution. Iron contamination will be evidenced by blue markings or spots, or a slight change or reduction in the white appearance of the dried paper when compared with the untreated paper.

5.2.2 Preparation

Immerse strips of a suitable paper, for example "wet strength" filter paper, in the sodium chloride or sodium chloride-gelatin solution (see 4.2.1) until they have been thoroughly wetted by the solution. Remove the strips and allow any excess solution to drain for about 1 min.

NOTES

3 A clean glass plate has been found useful for draining and holding the moistened test papers.

4 Application of the sodium chloride solution directly to the test surface produces diffuse rather than sharply defined pore sites. Therefore, the use of test paper which produces sharply defined pore sites is recommended.

5.2.3 Applying the test paper

Apply the wet test paper to the cleaned surface of the coating pressing it firmly into contact. It is essential that complete contact be made by the moistened test paper over the entire area under test in order that the sodium chloride solution may react with any exposed base metal. Allow the test paper to remain in contact for 10 min. If the paper becomes dry during the test, moisten it with additional solution taking care not to move it.

NOTE 5 For irregular or small surfaces it may be useful to cut the test paper to the shape of the area to be tested.

5.2.4 Test paper development

Remove the paper from the test surface and immediately place it in the potassium hexacyanoferrate(III) solution (see 4.2.2) to allow the blue solution to develop at the discontinuity sites.

5.3 Test paper examination

Examine the developed test paper surface for evidence of discontinuities. Sharply defined blue markings or spots are produced on the paper wherever the base steel or iron was exposed due to discontinuities in the coating.

5.4 Discontinuity enumeration

Count the blue markings or spots on the test area without the aid of magnification.

NOTE 6 For large test areas a plastic template (see 4.3) may be placed over the test paper, after development, and used for counting discontinuities.

5.5 Retest

If necessary, the same area may be retested. It is essential that the coating surface be thoroughly rinsed with hot analytical grade water (see 4.1) to remove any traces of residual gelatinized chloride reagent which may contain reaction products from the previous test. After cleaning, the part shall be thoroughly dried in order to avoid smudging any discontinuity indications produced during retest.

6 Expression of results

Express the porosity on the test area as an average value of X pores/cm^2 or as the maximum count of pores/cm^2 using a 10 mm by 10 mm counting template.

NOTE 7 Given that pass-fail criteria vary with the coating specification and the product specification, as well as with the thickness of coating used, they are not specified here.

7 Test report

The test report shall contain the following information:

a) the number of this International Standard;

b) the area of surface tested;

c) a reference to the relevant coating or product standard;

d) the result, expressed as in clause 6.

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