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Metallic coatings — Copper-accelerated acetic acid salt spray test (CASS test)

Revêtements métalliques — Essai au brouillard salin cupro-acétique (Essai CASS)

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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It has been approved by the Member Bodies of the following countries :

ISO 3770:1976

Australia	Israel	Spain
Austria	Italy	Sweden
Chile	Japan	Switzerland
Czechoslovakia	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.A.
Hungary	Romania	
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No Member Body expressed disapproval of the document.

Metallic coatings — Copper-accelerated acetic acid salt spray test (CASS test)

0 INTRODUCTION

There is seldom a direct relation between resistance to the action of salt spray and resistance to corrosion in other media because several factors influencing the progress of corrosion, such as the formation of protective films, vary greatly with the conditions encountered. Therefore, the results obtained in the test should not be regarded as a direct guide to the corrosion resistance of the tested coatings in all environments where these coatings may be used. Also, performance of different coatings in the test should not be taken as a direct guide to the relative corrosion resistance of these coatings in service.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the apparatus and procedure to be used in conducting the copper-accelerated acetic acid salt spray test (CASS test) for assessment of the quality of metallic and related coatings made in accordance with the requirements of coating or product specifications.

This test was devised for assessing the quality of decorative coatings of copper + nickel + chromium or nickel + chromium; it has also been found suitable for testing anodic oxide coatings on aluminium. The test should be used only for testing such coatings unless otherwise specified. This test is not reliable for comparing copper + nickel + chromium coatings with nickel + chromium coatings, because the accelerating effect of copper corrosion products from copper + nickel + chromium coatings is masked by the effect of copper ions present in the test solution.

This International Standard does not specify the type of test specimen, the exposure periods to be used for a particular product or the interpretation of results. Such details are provided in the appropriate coating or product specifications.

2 TEST SOLUTION

2.1 The test solution shall be prepared by dissolving sodium chloride in distilled or de-ionized water to produce a concentration of 50 ± 5 g/l. The sodium chloride shall be white and shall give a colourless solution in water. It shall be substantially free from copper and nickel and shall contain not more than 0,1 % of sodium iodide and not more than 0,4 % of total impurities calculated for dry

salt. If the pH of the solution as prepared is outside the range 6,0 to 7,0 the presence of undesirable impurities in the salt or the water or both should be investigated.

2.2 A sufficient amount of copper(II) chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) shall be dissolved in the salt solution to produce a concentration of $0,26 \pm 0,02$ g/l (equal to $0,205 \pm 0,015$ g/l CuCl_2).

2.3 A sufficient amount of glacial acetic acid shall be added to the salt solution to ensure that the pH of samples of sprayed solution collected in the test cabinet (see 3.1.4) will be between 3,1 and 3,3. If the pH of the solution as initially prepared is 3,0 to 3,1, the pH of the sprayed solution is likely to be within the specified limits. All pH values shall be measured electrometrically at 25 °C, but a short-range pH paper which can be read in increments of 0,1 pH unit or less and which has been calibrated against electrometric measurements may be used in routine checks. Any necessary correction shall be made by additions of solutions of glacial acetic acid or sodium hydroxide of analytical grade.

2.4 The solution shall be filtered before it is placed in the reservoir of the apparatus, in order to remove any solid matter which might block the apertures of the spraying devices.

3 APPARATUS

3.1 The apparatus shall comprise the following components :

3.1.1 A spray cabinet, made of, or lined with, material resistant to corrosion by the sprayed solution. The cabinet shall have a capacity of not less than 0,2 m³ and preferably not less than 0,4 m³ since, with smaller volumes, difficulties are experienced in ensuring even distribution of spray. The upper parts shall be so shaped that drops of sprayed solution accumulated on them do not fall on specimens being tested.

The size and shape of the cabinet shall be such that the quantity of solution collected on the cabinet is within the limits stated in 6.2.

3.1.2 Means of supplying and controlling heat, adequate to maintain the cabinet and its contents at the specified temperature (see 6.1). The temperature shall be controlled by a thermostat element placed either within the cabinet at least 100 mm from the walls or in a water jacket on the cabinet. In either case the thermometer, capable of being read from the outside, shall be placed within the cabinet at least 100 mm from the walls.

3.1.3 Means for spraying the salt solution, comprising a supply of clean air of controlled pressure and humidity, a reservoir to contain the solution to be sprayed and one or more atomizers made of inert material.

The compressed air supply to the atomizers shall be passed through a filter to remove all traces of oil or solid matter and shall be at a pressure of 70 to 170 kPa¹⁾. In order to prevent evaporation of water from the sprayed droplets the air shall be humidified before entering the atomizer by passage through a saturation tower containing water at a temperature several degrees higher than that of the cabinet. The appropriate temperature depends on the pressure used and on the type of atomizer nozzle and shall be adjusted so that the rate of collection of spray in the cabinet and the concentration of the collected spray are kept within the specified limits (see 6.2).

The reservoir to contain the solution to be sprayed shall be a tank made of material resistant to the solution and shall be provided with means of maintaining a constant level of solution in the reservoir.

The atomizers shall be made of inert material, for example glass or plastics material. Baffles may be used to prevent direct impingement of spray on test specimens and the use of adjustable baffles is helpful in obtaining uniform distribution of spray throughout the cabinet.

3.1.4 At least two suitable collecting devices, which shall be funnels of glass or other chemically inert material with the stems inserted into graduated cylinders or other containers of the same type. Funnels with a diameter of 100 mm have a collecting area of approximately 80 cm². The collecting devices shall be placed in the zone of the cabinet where the test specimens are placed, one close to an inlet of spray and one remote from an inlet. They shall be so placed that only spray and not liquid falling from specimens or from part of the cabinet is collected.

3.2 If the equipment has been used for a spray test or for any other purpose with solution differing from that specified for the test to be carried out, it shall be thoroughly cleaned before use.

4 TEST SPECIMENS

4.1 The number and type of test specimens, their shape and their dimensions shall be selected according to the specification covering the coating or product being tested. When not so specified, details concerning the specimens shall be mutually agreed between the interested parties.

4.2 The specimens shall be thoroughly cleaned before testing. The method employed shall depend on the nature of the surface and of the contaminants and shall not include the use of any abrasives or solvents which may attack the surface of the specimens.

Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

4.3 If test specimens are cut from a larger coated article, the cutting shall be carried out in such a way that the coating is not damaged in the area adjacent to the cut. Unless otherwise specified, the cut edges shall be adequately protected by coating them with a suitable medium, stable under the conditions of the test, such as paint, wax or adhesive tape.

5 METHOD OF EXPOSURE OF TEST SPECIMENS

5.1 The specimens shall be so placed in the cabinet that they are not in the direct line of travel of spray from the atomizer. Baffles may be used to prevent direct impact of the sprayed solution on the specimens.

5.2 The angle at which the sample is exposed in the cabinet is very important. The surface shall, in principle, be flat and placed in the cabinet facing upwards at an angle as close as possible to 20° to the vertical. This angle shall, in all cases, be within the limits 15° to 30°.

In the case of irregular surfaces, for example entire components, these limits shall be adhered to as closely as possible.

5.3 The specimens shall be so arranged that they do not come into contact with one another or with the cabinet and that surfaces to be tested are exposed to free circulation of spray. Specimens may be placed at different levels within the cabinet as long as solution cannot drop from specimens or their support at one level onto other specimens placed below.

5.4 The support for the specimens shall be made of inert non-metallic material such as glass, plastics or suitably coated wood. If it is necessary to suspend test specimens, the material used shall on no account be metallic and shall be synthetic fibre, cotton thread or other inert insulating material.

6 OPERATING CONDITIONS

6.1 The temperature inside the cabinet shall be 50 ± 2 °C with the minimum possible fluctuation throughout the cabinet during the test.

6.2 The solution collected in each of the collecting devices (see 3.1.4) shall have a sodium chloride concentration of 50 ± 10 g/l and a pH value in the range 3,1 to 3,3 (see 2.3).

1) 1 kPa = 1 kN/m² ≈ 0,01 atm

The average rate of collection of solution in each device measured over a minimum period of 24 h shall be 1 to 2 ml/h for a horizontal collecting area of 80 cm².

6.3 Test solution which has been sprayed shall not be re-used.

NOTE — In order to compare test conditions from one laboratory to another or from one day to another, a calibration may be made by exposing nickel panels, having dimensions 100 mm X 75 mm, to the spray at each of the four bottom corners of the cabinet. The loss of mass of such panels, treated as described in the annex, should be from 35 to 70 mg in a test period of 24 h.

7 DURATION OF TESTS

7.1 The period of test shall be as designated by the specifications covering the coating or product being tested. When not specified, it shall be mutually agreed by the parties interested.

Recommended periods of exposure are 2 h – 6 h – 24 h – 48 h – 96 h – 240 h – 480 h – 720 h.

7.2 Spraying shall not be interrupted during the prescribed test period. The cabinet shall be opened only for brief visual inspections of the test specimens in position and for replenishing the salt solution in the reservoir if such replenishment cannot be carried out from outside the cabinet.

7.3 If the end-point of the test depends on the appearance of the first sign of corrosion, the specimens will need to be inspected frequently. For this reason such specimens shall not be tested together with other specimens requiring tests of predetermined duration.

7.4 A periodic visual examination of specimens under test for a predetermined period may be made but the surfaces under test shall not be disturbed and the period for which the cabinet is open shall be the minimum necessary to observe and record any visible changes.

8 CLEANING OF SPECIMENS AFTER TEST

At the end of the test period, remove the specimens from the cabinet. Allow the specimens to dry for 0,5 to 1 h before rinsing, in order to reduce the risk of removing corrosion products. Before they are examined, carefully remove residues of the spray solution from their surfaces. A suitable method is to rinse or dip the specimens gently in clean running water at a temperature not exceeding 40 °C and then to dry them immediately in a stream of compressed air at a pressure not exceeding 200 kPa at a distance of approximately 300 mm.

9 EVALUATION OF RESULTS

Many different criteria for the evaluation of the results of the test may be applied to meet particular requirements, for example change in mass, alteration revealed by micro-graphic examination or change in mechanical properties.

Usually the appropriate criteria will be indicated in the specification for the coating or product tested. For most routine applications of the test, only the following need to be considered :

- a) appearance after test;
- b) appearance after removing superficial corrosion products;
- c) the number and distribution of corrosion defects, i.e. pits, cracks, blisters, etc.; these may conveniently be assessed by methods such as that described in ISO 1462, *Metallic coatings – Coatings other than those anodic to the basis metal – Accelerated corrosion tests – Method for evaluation of the results*;
- d) the time elapsing before the appearance of the first sign of corrosion.

10 TEST REPORT

10.1 The test report shall indicate the outcome of the test according to the criteria for evaluation of results prescribed for the test. The result obtained for each specimen tested and, when appropriate, the average result for a group of replicate test specimens shall be reported. The report, may, if required, be accompanied by photographic records of the tested specimens.

10.2 The report shall contain information about the test procedure. This information may vary according to the purposes of the test and to the directions prescribed for it but a general list of the details likely to be required is as follows :

- a) the description of the coating or product tested;
- b) dimensions and shape of the test specimen and the nature of the surface tested;
- c) preparation of the test specimen, including any cleaning treatment applied and any protection given to edges or other special areas;
- d) known characteristics of any coating, with an indication of the surface finish;
- e) the number of test specimens subjected to the test representing each coating or product;
- f) the method used to clean test specimens after test with, when appropriate, an indication of the loss in mass resulting from the cleaning operation;
- g) the angle at which the tested surfaces were inclined;
- h) the test temperature;
- i) the duration of test;
- j) the properties of any test panels placed in the cabinet expressly to check the correctness of the operating conditions and the results obtained with them.

ANNEX

CALIBRATION OF TEST CONDITIONS

A.1 GENERAL

This annex describes the method for evaluating the corrosive effect of the conditions produced in the spray test cabinet. The results obtained permit a comparison to be made of the test conditions obtained in different laboratories or in the same laboratory at different times.

A.2 TEST PANELS

The test panels shall be cut from commercially pure nickel sheet containing not less than 99,0 % nickel and not more than 0,15 % carbon, 0,01 % sulphur, 0,4 % iron and 0,25 % copper. A suitable sheet thickness is approximately 1 mm and the panel dimensions shall be 100 mm × 75 mm.

Panels to be used for the first time shall be cleaned by the general procedure specified in 4.2 and shall then be immersed for 2 min at a temperature of 21 to 24 °C in a solution prepared by mixing 1 volume of concentrated hydrochloric acid with 4 volumes of distilled water, rinsed in warm flowing water and dried in an oven at a temperature of 105 °C, cooled to room temperature and weighed. Panels used in the test may be re-used. The procedure for removing corrosion products at the end of a test will produce a satisfactory surface for a subsequent test.

A.3 ARRANGEMENT OF PANELS

The weighed panels shall be placed in the test cabinet with the longer edges inclined at 30° to the vertical with the upper surface facing the general direction of the incoming spray. Supports for the panels shall be made of or completely covered by inert materials such as plastics. The upper edges of the panels shall be at the same level as the tops of the spray collectors.

A.4 DETERMINATION OF MASS LOSS

After exposure to test for 24 h the panels shall be rinsed in cold water to remove spray residues. The corrosion products shall then be removed from each panel by immersing it for 2 min at a temperature of 21 to 24 °C in a solution prepared by mixing 1 volume of concentrated hydrochloric acid with 4 volumes of distilled water. Tongs used to hold the panels shall be of nickel or Monel metal or shall have their contact areas covered with inert material. Each panel shall then be washed in water with a final rinse in flowing water at a temperature of approximately 40 °C, dried in an oven at a temperature of 105 °C, cooled to room temperature and again weighed.

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