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Corrosion tests in artificial atmospheres — Salt spray tests

Essais de corrosion en atmosphères artificielles — Essais aux brouillards salins

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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 9227 was prepared by Technical Committee ISO/TC 156, Corrosion of metals and alloys.

This third edition cancels and replaces the second edition (ISO 9227:2006), of which it constitutes a minor revision.

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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 test results should not be regarded as a direct guide to the corrosion resistance of the tested metallic materials in all environments where these materials might be used. Also, the performance of different materials during the test should not be taken as a direct guide to the corrosion resistance of these materials in service.

Nevertheless, the method described gives a means of checking that the comparative quality of a metallic material, with or without corrosion protection, is maintained.

Salt spray tests are generally suitable as corrosion protection tests for rapid analysis for discontinuities, pores and damage in organic and inorganic coatings. In addition, for quality control purposes, comparison can be made between specimens coated with the same coating. As comparative tests, however, salt spray tests are only suitable if the coatings are sufficiently similar in nature.

It is often not possible to use results gained from salt spray testing as a comparative guide to the long-term behaviour of different coating systems, since the corrosion stress during these tests differs significantly from the corrosion stresses encountered in practice.

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Corrosion tests in artificial atmospheres — Salt spray tests

1 Scope

This International Standard specifies the apparatus, the reagents and the procedure to be used in conducting the neutral salt spray (NSS), acetic acid salt spray (AASS) and copper-accelerated acetic acid salt spray (CASS) tests for assessment of the corrosion resistance of metallic materials, with or without permanent or temporary corrosion protection.

It also describes the method employed to evaluate the corrosivity of the test-cabinet environment.

It does not specify the dimensions of test specimens, the exposure period to be used for a particular product, or the interpretation of results. Such details are provided in the appropriate product specifications.

The salt spray tests are particularly useful for detecting discontinuities, such as pores and other defects in certain metallic, organic, anodic oxide and conversion coatings.

The neutral salt spray test is the test method in which a 5 % sodium chloride solution in the pH range from 6,5 to 7,2 is atomized under a controlled environment. It particularly applies to:

- metals and their alloys,
- metallic coatings (anodic and cathodic), DARD PREVIEW
- conversion coatings,

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anodic oxide coatings, and

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organic coatings on metallic materials.
 organic coatings on metallic materials.

The acetic acid salt spray test is the test method in which a 5 % sodium chloride solution with the addition of glacial acetic acid in the pH range from 3,1 to 3,3 is atomized under a controlled environment. It is especially useful for testing decorative coatings of copper + nickel + chromium, or nickel + chromium. It has also been found suitable for testing anodic coatings on aluminum.

The copper-accelerated acetic acid salt spray test is the test method in which a 5 % sodium chloride solution with the addition of copper chloride and glacial acetic acid in the pH range from 3,1 to 3,3 is atomized under a controlled environment. It is useful for testing decorative coatings of copper + nickel + chromium, or nickel + chromium. It has also been found suitable for testing anodic coatings on aluminum.

The salt spray methods are all suitable for checking that the comparative quality of a metallic material, with or without corrosion protection, is maintained. They are not intended to be used for comparative testing as a means of ranking different materials relative to each other with respect to corrosion resistance.

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 1514, Paints and varnishes — Standard panels for testing

ISO 2808, Paints and varnishes — Determination of film thickness

ISO 3574, Cold-reduced carbon steel sheet of commercial and drawing qualities

ISO 8407, Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens

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ISO 17872, Paints and varnishes — Guidelines for the introduction of scribe marks through coatings on metallic panels for corrosion testing

3 Test solutions

3.1 Preparation of the sodium chloride solution

Dissolve a sufficient mass of sodium chloride in distilled or deionized water with a conductivity not higher than 20 μ S/cm at 25 °C \pm 2 °C to produce a concentration of 50 g/l \pm 5 g/l. The sodium chloride concentration of the sprayed solution collected shall be 50 g/l \pm 5 g/l. The specific gravity range for a 50 g/l \pm 5 g/l solution is 1,029 to 1,036 at 25 °C.

The sodium chloride shall contain less than 0,001 % mass fraction of copper and less than 0,001 % mass fraction of nickel, as determined by atomic absorption spectrophotometry or another analytical method of similar sensitivity. It shall not contain more than 0,1 % of a mass fraction of sodium iodide, or more than 0,5 % of a mass fraction of total impurities calculated for dry salt.

NOTE If the pH of the prepared solution at 25 $^{\circ}$ C \pm 2 $^{\circ}$ C is outside the range 6,0 to 7,0, investigate the presence of undesirable impurities in the salt and/or the water.

3.2 pH adjustment

3.2.1 pH of the salt solution

Adjust the pH of the salt solution to the desired value on the basis of the pH of the sprayed solution collected. (standards.iteh.ai)

3.2.2 NSS test

Adjust the pH of the salt solution (3.1) so that the pH of the sprayed solution collected within the test cabinet (4.2) is 6,5 to 7,2 at $25 \,^{\circ}\text{C} \pm 2 \,^{\circ}\text{C}$. Check the pH using electrometric measurement or in routine checks, with a short-range pH paper, which can be read in increments or 0,3 pH units or less. Make any necessary corrections by adding hydrochloric acid, sodium hydroxide or sodium bicarbonate solution of analytical grade.

Possible changes in pH may result from loss of carbon dioxide from the solution when it is sprayed. Such changes can be avoided by reducing the carbon dioxide content of the solution by, for example, heating it to a temperature above 35 °C before it is placed in the apparatus, or by making the solution using freshly boiled water.

3.2.3 AASS test

Add a sufficient amount of glacial acetic acid to the salt solution (3.1) to ensure that the pH of samples of sprayed solution collected in the test cabinet (4.2) is between 3,1 and 3,3. If the pH of the solution initially prepared is 3,0 to 3,1, the pH of the sprayed solution is likely to be within the specified limits. Check the pH using electrometric measurement at 25 °C \pm 2 °C, or in routine checks, with a short-range pH paper which can be read in increments of 0,1 pH units or less. Make any necessary corrections by adding glacial acetic acid or sodium hydroxide of analytical grade.

3.2.4 CASS test

Dissolve a sufficient mass of copper(II) chloride dihydrate (CuCl₂·2H₂O) in the salt solution (3.1) to produce a concentration of 0,26 g/I \pm 0,02 g/I [equivalent to (0,205 \pm 0,015) g of CuCl₂ per litre].

Adjust the pH using the procedures described in 3.2.3.

3.3 Filtration

If necessary, filter the solution before placing it in the reservoir of the apparatus, to remove any solid matter which might block the apertures of the spraying device.

4 Apparatus

4.1 Component protection

All components in contact with the spray or the test solution shall be made of, or lined with, materials resistant to corrosion by the sprayed solution and which do not influence the corrosivity of the sprayed test solutions.

4.2 Spray cabinet

The cabinet shall be such that the conditions of homogeneity and distribution of the spray are met. The upper parts of the cabinet shall be designed so that drops of sprayed solution formed on its surface do not fall on the specimens being tested.

The size and shape of the cabinet shall be such that the collection rate of solution in the cabinet is within the limits specified in Table 2, measured as specified in 8.3.

Preference shall be given to apparatus that has a means for properly dealing with fog after the test, prior to releasing it from the building for environmental conservation, and for drawing water prior to discharging it to the drainage system.

NOTE A schematic diagram of one possible design of spray cabinet is shown in Annex A.

4.3 Heater and temperature control

An appropriate system maintains the cabinet and its contents at the specified temperature (see Table 2). The temperature shall be measured at least 100 mm from the walls.

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4.4 Spraying device

The device for spraying the salt solution comprises a supply of clean air, of controlled pressure and humidity, a reservoir to contain the solution to be sprayed, and one or more atomizers.

The compressed air supplied to the atomizers shall be passed through a filter to remove all traces of oil or solid matter, and the atomizing pressure shall be at an overpressure of 70 kPa $^{1)}$ to 170 kPa. The pressure should be 98 kPa \pm 10 kPa.

NOTE Atomizing nozzles can have a "critical pressure" at which an abnormal increase in the corrosiveness of the salt spray occurs. If the "critical pressure" of a nozzle has not been established with certainty, control of fluctuations in the air pressure within ± 0.7 kPa, by installation of a suitable pressure regulator valve, minimizes the possibility that the nozzle will be operated at its "critical pressure".

In order to prevent the evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer by passing through a saturation tower containing hot distilled water or deionized water at a temperature 10 °C above 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 8.3). In Table 1, guiding values are given for the hot-water temperature in the saturation tower at different pressures. The level of the water shall be maintained automatically to ensure adequate humidification.

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¹⁾ $1 \text{ kPa} = 1 \text{ kN/m}^2 = 0.01 \text{ atm} = 0.01 \text{ bar} = 0.145 \text{ psi}.$

Table 1 — Guiding values for the temperature	of the hot water in the saturation tower
----------------------------------------------	------------------------------------------

Atomizing overpressure	Guiding values for the temperature, in °C, of the hot water in the saturation tower when performing the different salt spray tests	
kPa	Neutral salt spray (NSS) and acetic acid salt spray (AASS)	Copper-accelerated acetic acid salt spray (CASS)
70	45	61
84	46	63
98	48	64
112	49	66
126	50	67
140	52	69

The atomizers shall be made of inert material. Baffles may be used to prevent direct impact of the spray on the test specimens, and the use of adjustable baffles is helpful in obtaining uniform distribution of the spray within the cabinet. For this purpose, a dispersion tower equipped with an atomizer may also be helpful. The level of the salt solution in the salt reservoir shall be maintained automatically to ensure uniform spray delivery throughout the test.

4.5 Collecting devices

At least two suitable collecting devices shall be available, consisting of funnels made of chemically inert material, with the stems inserted into graduated cylinders or other similar containers. Suitable funnels have a diameter of 100 mm, which corresponds to 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 placed so that only mist, and not liquid falling from specimens or from parts of the cabinet, is collected.

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4.6 Re-use

If the cabinet has been used once for an AASS or CASS test, or has been used for any other purpose with a solution differing from that specified for the NSS test, it shall not be used for the NSS test.

It is nearly impossible to clean a cabinet that was once used for AASS or CASS testing so that it can be used for an NSS test. However, in such circumstances, the equipment shall be thoroughly cleaned and checked using the method described in Clause 5, ensuring in particular that the pH of the collected solution is correct throughout the entire spraying period. After this procedure, the specimens to be tested are placed in the cabinet.

5 Method for evaluating cabinet corrosivity

5.1 General

To check the reproducibility and repeatability of the test results for one piece of apparatus, or for similar items of apparatus in different laboratories, it is necessary to verify the apparatus at regular intervals as described in 5.2 to 5.4.

NOTE During permanent operation, a reasonable time period between two checks of the corrosivity of the apparatus is generally considered to be 3 months.

To determine the corrosivity of the tests, reference-metal specimens made of steel shall be used.

As a complement to the reference-metal specimens made of steel, high-purity zinc reference-metal specimens may also be exposed in the tests in order to determine the corrosivity against this metal as described in Annex B.

5.2 NSS test

5.2.1 Reference specimens

To verify the apparatus, use four or six reference specimens of 1 mm \pm 0,2 mm thickness and 150 mm \times 70 mm, of CR4-grade steel in accordance with ISO 3574 with an essentially faultless surface²⁾ and a matt finish (arithmetical mean deviation of the profile $Ra = 0.8 \ \mu m \pm 0.3 \ \mu m$). Cut these reference specimens from cold-rolled plates or strips.

Clean the reference specimens carefully, immediately prior to testing. Besides the specifications given in 6.2 and 6.3, cleaning shall eliminate all those traces (dirt, oil or other foreign matter) that could influence the test results.

Thoroughly clean the reference specimens with an appropriate organic solvent (such as a hydrocarbon with a boiling point between 60 °C and 120 °C) using a clean soft brush or an ultrasonic cleaning device. Carry out the cleaning in a vessel full of solvent. After cleaning, rinse the reference specimens with fresh solvent and then dry them.

Determine the mass of the reference specimens to ± 1 mg. Protect one face of the reference specimens with a removable coating, for example an adhesive plastic film. The edges of the reference test specimens may be protected by the adhesive tape as well.

5.2.2 Arrangement of the reference specimens

Position four steel reference specimens in four quadrants (if six specimens are available, place them in six different positions including four quadrants) in the zone of the cabinet where the test specimens are placed, with the unprotected face upwards, and at an angle of $20^{\circ} \pm 5^{\circ}$ from the vertical.

The support for the reference specimens shall be made of, or coated with, inert materials such as plastics. The lower edge of the reference specimens shall be level with the top of the salt spray collector. The test duration shall be 48 h.

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The test cabinet shall be filled with dummy specimens of inert materials, such as plastic or glass, during the verification procedure.

5.2.3 Determination of mass loss (mass per area)

At the end of the test, immediately take the reference specimens out of the test cabinet and remove the protective coating. Remove the corrosion products by mechanical and chemical cleaning, as described in ISO 8407. For chemical cleaning, use a solution with a mass fraction of 20 % of diammonium citrate [(NH₄)₂HC₆H₅O₇] (recognized analytical grade) in water for 10 min at 23 °C.

After each stripping, thoroughly clean the reference specimens at ambient temperature with water, then with ethanol, followed by drying.

Weigh the reference specimens to the nearest 1 mg. Divide the determined mass loss by the area of the exposed surface area of the reference specimen in order to assess the metal mass loss per square metre of the reference specimen.

It is recommended that freshly prepared solution be used during each procedure for the removal of corrosion products.

NOTE The corrosion products can also be removed by chemical cleaning as described in ISO 8407, and by using a solution with a volume fraction of 50 % hydrochloric acid ($\rho_{20} = 1,18$ g/ml), of recognized analytical grade, in water, the latter also containing 3,5 g/l of hexamethylene tetramine as a corrosion inhibitor.

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^{2) &}quot;Essentially faultless" means free from pores, marks, scratches and any light colouration.