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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

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

It cancels and replaces International Standards ISO 3768:1976, ISO 3769:1976 and ISO 3770:1976.

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

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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 may 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.

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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 corrosion protection 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, anodic oxide and conversion coatings.

The neutral salt spray test applies to

- metals and their alloys;
- certain metallic coatings (anodic and cathodic)¹⁾;
- certain conversion coatings¹⁾;
- certain anodic oxide coatings¹⁾;
- organic coatings on metallic materials.

The acetic acid salt spray test is especially useful for testing decorative coatings of copper + nickel +

chromium or nickel + chromium. It has also been found suitable for testing anodic oxide coatings on aluminium.

The copper-accelerated acetic acid salt spray test is useful for testing decorative coatings of copper + nickel + chromium or nickel + chromium; it has also been found suitable for testing anodic coatings on aluminium.²⁾

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1462:1973, *Metallic coatings — Coatings other than those anodic to the basis metal — Accelerated corrosion tests — Method for the evaluation of the results.*

ISO 3574:1986, *Cold-reduced carbon steel sheet of commercial and drawing qualities.*

ISO 4540:1980, *Metallic coatings — Coatings cathodic to the substrate — Rating of electroplated test specimens subjected to corrosion tests.*

ISO 6372-1:1989, *Nickel and nickel alloys — Terms and definitions — Part 1: Materials.*

1) See annex B.

2) Attention is drawn to the fact that no satisfactory basis for comparison can be derived from this test with regard to the respective quality of nickel + chromium coatings and copper + nickel + chromium coatings, because the reagent used contains a copper ion which promotes corrosion in the presence of nickel but is without influence on copper.

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 $20 \mu\text{S}/\text{cm}$ at $25 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ to produce a concentration of $50 \text{ g/l} \pm 5 \text{ g/l}$. The sodium chloride concentration of the sprayed solution collected shall be $50 \text{ g/l} \pm 5 \text{ g/l}$. The specific gravity range for a $50 \text{ g/l} \pm 5 \text{ g/l}$ solution is 1,025 5 to 1,040 0 at $25 \text{ }^{\circ}\text{C}$.

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

NOTE 1 If the pH of the prepared solution, measured at $25 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$, 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

Adjust the pH of the salt solution to the desired value on the basis of the pH of the sprayed solution collected.

3.2.1 NSS test

Adjust the pH of the salt solution (3.1) so that the pH of the sprayed solution collected within the test cabinet (4.1) is between 6,5 to 7,2. Check the pH using electrometric measurement at $25 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$, or, in routine checks, with a short-range pH paper which can be read in increments of 0,3 pH units or less. Make any necessary correction by adding hydrochloric acid or sodium hydroxide solution of analytical grade.

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

3.2.2 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 (4.1) is 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. Check the pH using electrometric measurement at $25 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$, 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 correction by adding glacial acetic acid or sodium hydroxide of analytical grade.

3.2.3 CASS test

Dissolve a sufficient mass of copper(II) chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) in the salt solution (3.1) to produce a concentration of $0,26 \text{ g/l} \pm 0,02 \text{ g/l}$ [equivalent to $(0,205 \pm 0,015) \text{ g}$ of CuCl_2 per litre].

Adjust the pH using the procedures described in 3.2.2.

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

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. The apparatus shall include the following components.

4.1 Spray cabinet

The cabinet shall have a capacity of not less than $0,2 \text{ m}^3$ and preferably of not less than $0,4 \text{ m}^3$ since, with smaller volumes, difficulties were experienced in ensuring the even distribution of spray. For large-capacity cabinets, it is necessary to ensure 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 8.2.

NOTE 2 A schematic diagram of one possible design of spray cabinet is shown in annex A.

4.2 Heater and temperature control

An appropriate system maintains the cabinet and its contents at the specified temperature (see 8.1). The temperature shall be measured at least 100 mm from the walls.

4.3 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 shall be at an absolute pressure of 70 kPa³⁾ to 170 kPa.

NOTE 3 Atomizing nozzles may 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 $\pm 0,7 \text{ kN/m}^2$ ($\pm 0,1 \text{ psi}$), 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 evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer, by passage through a saturation tower containing hot water at a temperature several degrees Celsius 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 8.2). The level of the water must be maintained automatically to ensure adequate humidification.

The atomizers shall be made of inert material, for example glass or plastics materials. Baffles may be used to prevent direct impact of spray on the test specimens and the use of adjustable baffles is helpful in obtaining uniform distribution of the spray within the cabinet. The level of the salt solution in the salt reservoir shall be maintained automatically to ensure uniform spray delivery throughout the test.

4.4 Collecting devices

At least two suitable collecting devices shall be available, consisting of funnels made of glass or other chemically inert material, with the stems inserted into graduated cylinders or other similar containers. 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 placed so that only spray, and not liquid falling from specimens or from parts of the cabinet, is collected.

3) $1 \text{ kPa} \approx 1 \text{ kN/m}^2 \approx 0,01 \text{ atm}$

4) "Practically faultness" means free from pores, slights, marks, scratches, and any light coloration.

4.5 Re-use

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

The equipment shall then be operated for a minimum period of 24 h and the pH of the collected solution measured to ensure that pH is correct throughout the entire spraying period, before any specimens are placed in the chamber.

5 Method of evaluation of the corrosivity of the chamber

To check the reproducibility of the test results for one piece apparatus or for similar items of apparatus in different laboratories, it is necessary to verify the apparatus at regular intervals as described in 5.1.1 to 5.1.3.

5.1 NSS test

5.1.1 Reference specimens

To verify the apparatus, use four reference specimens $1 \text{ mm} \pm 0,2 \text{ mm}$ thick and $50 \text{ mm} \times 80 \text{ mm}$, of CR4 grade steel according to ISO 3574, with a practically faultless surface⁴⁾, and a mat finish (arithmetic mean deviation of the profile $R_a = 1,3 \mu\text{m} \pm 0,4 \mu\text{m}$). Cut these reference specimens from cold-rolled plates or strips.

Clean the reference specimens carefully immediately before testing. Besides the specifications given in 6.2 and 6.3, cleaning shall eliminate all traces of dirt, oil or other foreign matter capable of influencing the test results.

Use one of the following methods:

- Clean the reference specimens by vapour degreasing with a chlorinated hydrocarbon. Use three successive treatments of 1 min each, with an interval of at least 1 min between successive treatments.
- Thoroughly clean the reference specimens with an appropriate organic solvent (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, then dry them.

- c) Other cleaning methods may be used, after agreement between the interested parties, subject to the result being comparable.

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.

5.1.2 Arrangement of the reference specimens

Position the four reference specimens in four different quadrants in the test apparatus, with the unprotected face upwards, and at an angle of $20^\circ \pm 5^\circ$ from the vertical.

NOTE 4 Other angles may be recommended within specifications (e.g. $30^\circ \pm 5^\circ$).

The support for the reference specimens shall be made of, or coated with, inert materials such as plastics. The upper edge of the reference specimens shall be level with the top of the salt spray collector. The test duration is 96 h.

5.1.3 Determination of mass loss

At the end of the test, remove the protective coating. Remove the corrosion products by immersion in cleaning solution of hydrochloric acid ($\rho_{20} = 1,18$ g/ml), recognized analytical grade, 50 % (V/V), in water, inhibited by 3,5 g of hexamethylene tetramine per litre.

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

Weigh the reference specimens to the nearest 1 mg and calculate the mass loss in grams per square metre.

5.1.4 Checking of NSS apparatus operation

The operation of the test apparatus is satisfactory if the mass loss of each reference specimen is $140 \text{ g/m}^2 \pm 40 \text{ g/m}^2$.

5.2 AASS test

No experimental procedure exists to determine the corrosivity of the test chamber for AASS in this International Standard.

5) If necessary, the exposure time may be reduced to 6 h.

5.3 CASS test

5.3.1 Reference specimens

To verify the apparatus use four reference specimens $1 \text{ mm} \pm 0,2 \text{ mm}$ thick and $100 \text{ mm} \times 75 \text{ mm}$, made of nickel in accordance with the requirements of ISO 6372-1:1989, sub-clause 3.1. Cut these reference specimens from sheet metal.

Clean the reference specimens being used for the first time as specified in 6.2, then immerse them for 2 min, at a temperature of 21°C to 24°C , in a solution of 1 volume of concentrated hydrochloric acid and 4 volumes of distilled water. Rinse the reference specimens under warm running water and dry in an oven at a temperature of 105°C , then cool at room temperature and weigh. Reference specimens which have already been used may be used again. The procedure adopted to remove corrosion products at the end of the test shall provide a surface which is suitable for further testing.

Before testing, weigh the reference specimens to the nearest 1 mg. Protect one face of the reference specimens as described in 5.1.1.

5.3.2 Arrangement of the reference specimens

Position the four reference specimens in four different quadrants in the test apparatus, with the unprotected face upwards, and at an angle of $20^\circ \pm 5^\circ$ from the vertical (however, see 5.1.2, note 4). The support for the reference specimens shall be made of, or coated with, inert materials such as plastics. The upper edge of the reference specimens shall be level with the top of the salt spray collector.

5.3.3 Determination of mass loss (mass per area)

After a 24 h⁵⁾ exposure, rinse the reference specimens with cold water to remove any salt deposit. Eliminate the corrosion products by immersion for 2 min, at a temperature of $25^\circ \text{C} \pm 2^\circ \text{C}$, in a solution consisting of 1 volume of concentrated hydrochloric acid and 4 volumes of distilled water. The clamps used for holding the reference specimens shall be made of nickel or Monel and have their supporting parts coated with inert material. Rinse each specimen under running water at a temperature of $40^\circ \text{C} \pm 5^\circ \text{C}$, dry in an oven at a temperature of 105°C , then cool to room temperature and weigh again to the nearest 1 mg.

5.3.4 Checking of CASS apparatus operation

The test apparatus shall be considered to operate satisfactorily if the loss in mass of each specimen is $7,5 \text{ g/m}^2 \pm 2,5 \text{ g/m}^2$.

6 Test specimens

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

6.2 The test specimens shall be thoroughly cleaned before testing (see 5.1.1). The cleaning method employed shall depend on the nature of the material, its surface and 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 careless handling.

Specimens intentionally coated with protective organic films should not be cleaned prior to the test.

6.3 If the test specimens are cut from a larger coated article, 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 material, stable under the conditions of the test, such as paint, wax or adhesive tape.

7 Arrangement of the test specimens

7.1 The test specimens shall be placed in the cabinet so that they are not in the direct line of travel of spray from the atomizer.

7.2 The angle at which the surface of the test specimen is exposed in the cabinet is very important. The specimen 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.

7.3 The test specimens shall be arranged so that they do not come into contact with the cabinet and so that surfaces to be tested are exposed to free circulation of spray. The specimens may be placed at different levels within the cabinet, as long as the solution does not drip from specimens or their supports at one level onto other specimens placed below. However, for a new examination or for tests

with a total duration exceeding 96 h, location permutation of specimens is permitted⁶⁾.

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

8 Operating conditions

8.1 The temperature inside the spray cabinet should be $35\text{ °C} \pm 2\text{ °C}$ for neutral and acetic acid salt spray and $50\text{ °C} \pm 2\text{ °C}$ for copper-acetic acid salt spray, with the minimum possible fluctuation of temperature throughout the cabinet during the test.

8.2 The test shall begin after it has been confirmed that the collection rate and conditions are within the specified ranges, when the cabinet is filled with test specimens as planned.

8.3 The solution collected in each of the collecting devices (4.4) shall have a sodium chloride concentration of $50\text{ g/l} \pm 5\text{ g/l}$ and a pH value in the range of:

- 6,5 to 7,2 for neutral salt spray;
- 3,1 to 3,3 for acetic acid salt spray;
- 3,1 to 3,3 for copper-acetic acid salt spray.

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

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

8.5 During the test, any increase or decrease of cabinet pressure shall be avoided.

9 Duration of tests

9.1 The period of test shall be as designated by the specification covering the material or product being tested. When not specified, this period shall be agreed upon by the interested parties.

Recommended periods of exposure are 2 h - 6 h - 24 h - 48 h - 96 h - 168 h - 240 h - 480 h - 720 h - 1 000 h.

6) In this case, the number and the frequency of the permutations are left to the operator and are to be indicated in the test report.

9.2 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.

9.3 If the end-point of the test depends on the appearance of the first sign of corrosion, the test specimens shall be inspected frequently. For this reason this type of specimen shall not be tested with other specimens requiring tests of predetermined durations.

9.4 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.

10 Treatment of specimens after test

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

11 Evaluation of results

Many different criteria for the evaluation of the test results may be applied to meet particular requirements, for example:

- a) appearance after the test;
- b) appearance after removing superficial corrosion products;
- c) number and distribution of corrosion defects, i.e. pits, cracks, blisters, etc.; these may be assessed by methods described in ISO 1462 or ISO 4540;
- d) the time elapsing before the appearance of the first signs of corrosion;
- e) change in mass;
- f) alteration revealed by micrographic examination;
- g) change in mechanical properties.

NOTE 5 It is good engineering practice to define the appropriate criteria in the specification for a coating or a product to be tested.

12 Test report

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

12.2 The test report shall contain information about the test procedure. This information may vary according to the purpose 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) reference to this International Standard;
- b) type and purity of salt and water used;
- c) the description of the material or product tested;
- d) dimensions and shape of the test specimen and the nature and area of the surface tested;
- e) preparation of the test specimen, including any cleaning treatment applied and any protection given to edges or other special areas;
- f) known characteristics of any coating, with an indication of the surface area;
- g) the number of test specimens subjected to the test representing each material or product;
- h) the method used to clean test specimens after the test with, where appropriate, an indication of the loss in mass resulting from the cleaning operation;
- i) the angle at which the tested surfaces were inclined;
- j) the frequency and number of specimen location permutations, if any;
- k) the duration of the test and the results of any intermediate inspections;
- l) the properties of any reference specimens placed in the cabinet to check the stability of the operating conditions;
- m) test temperature;
- n) the volume of collected solution;

- o) pH of the test solution and the collected solution;
- p) density of the collected solution;
- q) any abnormality or incident occurring during the entire test procedure;
- r) intervals of inspection.

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Annex A (informative)

Schematic diagram of one possible design of spray cabinet

