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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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Contents

Page

Forev	word	v		
Intro	duction	vi		
1	Scope	1		
2	Normative references			
3	Terms and definitions			
4	Principle			
5	Test solutions 5.1 Preparation of the sodium chloride solution 5.2 pH adjustment 5.2.1 pH of the salt solution 5.2.2 Neutral salt spray (NSS) test 5.3 5.2.2	3 3 3 3 3		
	 5.2.3 Acetic acid salt spray (AASS) test 5.2.4 Copper-accelerated acetic acid salt spray (CASS) test 5.3 Filtration 	3 4 4		
6	Apparatus 6.1 Component protection 6.2 Spray cabinet 6.3 Heater and temperature control 6.4 Spraying device 6.5 Collecting devices 6.6 Re-use	4 4 4 4 4 5 5		
7	Method for evaluating cabinet corrosivity7.1General7.2Reference specimens7.3Arrangement of the reference specimens7.4Determination of mass loss (mass per area)7.5Satisfactory performance of cabinet	6 6 6 7 7		
8	Test specimens	7		
9	Arrangement of the test specimens	8		
10	Operating conditions	8		
11	Duration of tests	9		
12	Treatment of test specimens after test 12.1 General 12.2 Non-organic coated test specimens: metallic and/or inorganic coated 12.3 Organic coated test specimens 12.3.1 Scribed organic coated test specimens 12.3.2 Organic coated but not scribed test specimens	9 9 9 9 9 9		
13	Evaluation of results	10		
14	Test report	10		
Anne	ex A (informative) Example schematic diagram of one possible design of spray cabinet with means for optional treating fog exhaust and drain	12		
Anne	ex B (informative) Complementary method for evaluating cabinet corrosivity using zinc reference specimens	14		
Anne	ex C (normative) Preparation of specimens with organic coatings for testing	16		
Anne	ex D (normative) Required supplementary information for testing test specimens with organic coatings	17		

Bibliography 18

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see <u>www.iso.org/patents</u>).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: <u>www.iso.org/iso/foreword.html</u>.

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

This fourth edition cancels and replaces the third edition (ISO 9227:2012), which has been technically revised. The main technical changes are as follows:

- new definitions for reference material, reference specimen, test specimen and substitute specimen have been implemented:
- checking of the test apparatus during test operation has been made possible;
- <u>Clause 4</u> has been added, with some of its text moved from the scope;
- Clause 7 has been summarized.

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.

Different metallic substrates (metals) cannot be tested in direct comparison in accordance to their corrosion resistances in salt spray tests. Comparative testing is only applicable for the same kind of substrate.

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 document 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 or types 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 (NSS) test particularly applies to

- metals and their alloys,
- metallic coatings (anodic and cathodic),
- conversion coatings,
- anodic oxide coatings, and
- organic coatings on metallic materials.

The acetic acid salt spray (AASS) test is especially useful for testing decorative coatings of copper + nickel + chromium, or nickel + chromium. It has also been found suitable for testing anodic and organic coatings on aluminium.

The copper-accelerated acetic acid salt spray (CASS) test is useful for testing decorative coatings of copper + nickel + chromium, or nickel + chromium. It has also been found suitable for testing anodic and organic coatings on aluminium.

The salt spray methods are all suitable for checking that the 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 or as means of predicting long-term corrosion resistance of the tested material.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 4623-2:2016, Paints and varnishes — Determination of resistance to filiform corrosion — Part 2: Aluminium substrates

ISO 4628-1, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 1: General introduction and designation system

ISO 4628-2, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 2: Assessment of degree of blistering

ISO 4628-3, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 3: Assessment of degree of rusting

ISO 4628-4, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 4: Assessment of degree of cracking

ISO 4628-5, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 5: Assessment of degree of flaking

ISO 4628-8, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 8: Assessment of degree of delamination and corrosion around a scribe or other artificial defect

ISO 8044, Corrosion of metals and alloys — Basic terms and definitions

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

ISO 8993, Anodizing of aluminium and its alloys — Rating system for the evaluation of pitting corrosion — Chart method

ISO 10289, Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1

reference material

material with known test performance

3.2

reference specimen

portion of the *reference material* (3.1) that is to be exposed with the intention to check the reproducibility and repeatability of the test results for the test cabinet in use

3.3

test specimen

specific portion of the samples upon which the testing is to be performed

3.4

substitute specimen

specimen made of inert materials (such as plastic or glass) used for the substitute of a *test specimen* (3.3)

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4 Principle

The neutral salt spray (NSS) test is the test method in which a neutral 5 % sodium chloride solution is atomized under a controlled environment.

The acetic acid salt spray (AASS) test is the test method in which an acidified 5 % sodium chloride solution with the addition of glacial acetic acid is atomized under a controlled environment.

The copper-accelerated acetic acid salt spray (CASS) test is the test method in which an acidified 5 % sodium chloride solution with the addition of copper chloride and glacial acetic acid is atomized under a controlled environment.

5 Test solutions

5.1 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 ± 2 °C to produce a concentration of 50 g/l ± 5 g/l. The sodium chloride concentration of the sprayed solution collected shall be 50 g/l ± 5 g/l. The specific gravity range for a 50 g/l ± 5 g/l solution is 1,029 to 1,036 at 25 °C.

The sodium chloride shall not contain a mass fraction of the heavy metals copper (Cu), nickel (Ni) and lead (Pb) in total more than 0,005 %. It shall not contain a mass fraction of sodium iodide more than 0,1 % and a mass fraction of total impurities more than 0,5 %, calculated for dry salt.

NOTE Sodium chloride with anti-caking agents can act as corrosion inhibitors or accelerators. A useful sodium chloride salt grade is a grade named Ph. Eur/USP or JIS, ACS.

5.2 pH adjustment

5.2.1 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.

5.2.2 Neutral salt spray (NSS) test

Adjust the pH of the salt solution (5.1) so that the pH of the sprayed solution collected within the test cabinet (6.2) is 6,5 to 7,2 at 25 °C \pm 2 °C. Check the pH using electrometric measurement. Measurements of pH shall be done using electrodes suitable for measuring in weakly buffered sodium chloride solutions in deionized water. Make any necessary corrections by adding hydrochloric acid, sodium hydroxide or sodium bicarbonate solution of analytical grade.

NOTE Possible changes in pH can result from loss of carbon dioxide in 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.

5.2.3 Acetic acid salt spray (AASS) test

Add a sufficient amount of glacial acetic acid to the salt solution (5.1) to ensure that the pH of samples of sprayed solution collected in the test cabinet (6.2) is between 3,1 and 3,3 at 25 °C \pm 2 °C. 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. Measurements of pH shall be done using electrodes suitable for measuring in weakly buffered sodium chloride solutions in deionized water. Make any necessary corrections by adding glacial acetic acid, sodium hydroxide, or sodium bicarbonate of analytical grade.

5.2.4 Copper-accelerated acetic acid salt spray (CASS) test

Dissolve a sufficient mass of copper(II) chloride dihydrate (CuCl₂·2H₂O) in the salt solution (5.1) to produce a concentration of 0,26 g/l \pm 0,02 g/l [equivalent to (0,205 \pm 0,015) g of CuCl₂ per litre].

Adjust the pH using the procedures described in <u>5.2.3</u>.

5.3 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.

6 Apparatus

6.1 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.

The supports for the test specimen shall be constructed such that different substrate types do not influence each other. It shall also be constructed so that the supports themselves do not influence the test specimens.

6.2 Spray cabinet

The cabinet shall be such that the conditions of homogeneity and distribution of the spray are met. Due to the limited capacity of cabinets smaller than 0,4 m³, the effect of the loading of the cabinet on the distribution of the spray and temperature shall be carefully considered. The solution shall not be sprayed directly onto test specimens but rather spread throughout the cabinet so that it falls naturally down to them. The upper parts of the cabinet shall be designed so that drops of sprayed solution formed on its surface do not fall on the test 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 measured as specified in 10.3.

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 <u>Annex A</u> (see <u>Figure A.1</u> and <u>Figure A.2</u>).

6.3 Heater and temperature control

An appropriate system maintains the cabinet and its contents at the specified temperature (see <u>10.1</u>). The temperature shall be measured at least 100 mm from walls and radiant heat sources.

6.4 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 kPa¹) to 170 kPa. The pressure is typically 98 kPa \pm 10 kPa but can vary depending on the type of cabinet and atomizer used.

¹⁾ $1 \text{ kPa} = 1 \text{ kN/m}^2 = 0,01 \text{ atm} = 0,01 \text{ bar} = 0,145 \text{ psi}.$

In order to prevent the evaporation of water from the sprayed droplets (aerosol), the air shall be humidified before entering the atomizer by passing through a suitable humidifier. The humidified air shall be saturated such that the concentration of the fallout solution falls within the specifications of 5.1. The humidified air shall also be heated such that when mixed with the salt solution, there is no significant disturbance of the temperature in the cabinet. The appropriate temperature depends on the pressure used and on the type of atomizer nozzle. Temperature, pressure or humidification, or a combination thereof, shall be adjusted so that the rate of collection of the spray in the cabinet and the concentration of the collected spray are kept within the specified limits (see 10.3). A commonly used humidifier is the saturation tower, where temperature and pressure are controllable. Table 1 gives guiding values on temperature and pressure combinations for 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 test		
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	
160	53	70	
170	54	71	

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

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 salt solution supplied to the nozzle shall be kept stable to ensure a continuous and uniform fall out as described in 10.3. A stable level of spraying can be achieved by either controlling the level of salt solution in the reservoir or restricting the flow of salt solution to the nozzle such that a continuous spray is achieved.

Distilled or deionized water with a conductivity not higher than 20 μ S/cm at 25 °C ± 2 °C shall be used for humidification of spray air.

6.5 Collecting devices

At least two collecting devices shall be used to check the homogeneity of the spraying of the cabinet. Suitable funnels shall be made of chemically inert material, with the stems inserted into graduated cylinders or other similar containers and 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.

6.6 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 until a thorough cleaning procedure has been completed and the pH of collected solution has been verified

by the method in <u>5.2.2</u> and the corrosivity of the cabinet verified by the method in <u>Clause 7</u> to not be significantly affected by previous tests.

NOTE It is very difficult to clean a cabinet sufficiently that was once used for AASS or CASS testing so that it can be used for an NSS test.

7 Method for evaluating cabinet corrosivity

7.1 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 7.2 to 7.4.

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 specimens made of steel shall be used.

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

7.2 Reference specimens

To verify the apparatus, use at least four reference specimens of 1,0 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 $Ra = 0.8 \ \mu\text{m} \pm 0.3 \ \mu\text{m}$). Cut these reference specimens from cold-rolled plates or strips.

Clean the reference specimens carefully, immediately prior to testing. Besides the specifications given in 8.2 and 8.3, cleaning shall eliminate all those traces (dirt, oil or other foreign matter) that can 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 soft cloth, non-woven lint free cloth, that does not leave any remains 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 specimens may be protected by the adhesive tape as well.

7.3 Arrangement of the reference specimens

Position at least 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 in level with the top of the salt spray collector.

The cabinet should be verified during the testing of test specimens. If this is the case, great care shall be taken that the specimens do not affect each other. Otherwise, the cabinet shall be filled with substitute specimens to maintain the homogeneity of the cabinet. The verification procedure shall be performed using the same settings as for the test runs.

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

7.4 Determination of mass loss (mass per area)

At the end of the test with duration according to Table 2, 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. An option for chemical cleaning is the use of a solution with a mass fraction of 20 % of diammonium citrate $[(NH_4)_2HC_6H_5O_7]$ (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.

7.5 Satisfactory performance of cabinet

The cabinet has performed satisfactorily if the mass loss of steel reference specimen is within the allowed ranges given in <u>Table 2</u>.

Table 2 — Allowed range of mass loss of the steel reference specimens³⁾ during verification of the corrosivity of the cabinet

Test method	Test duration h	Allowed range of mass loss g/m^2
NSS	48	70 ± 20
AASS	24	40 ± 10
CASS	24	55 ± 15

NOTE See <u>Annex B</u> for use of zinc reference specimen.

8 Test specimens

8.1 The number and type of test specimens, their shape and their dimensions shall be selected in accordance with the specification for the material or product being tested. When not specified, these parameters shall be mutually agreed between the interested parties. Unless otherwise specified or agreed, test specimens with an organic coating to be tested shall be made from burnished steel complying with ISO 1514, and of approximate dimensions 150 mm × 100 mm × 1 mm. <u>Annex C</u> describes how test specimens with organic coatings are to be prepared for testing. <u>Annex D</u> gives supplementary information needed for testing test specimens with organic coatings.

8.2 The test specimens shall be thoroughly cleaned before testing, if not otherwise specified. 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 test specimens are not recontaminated after cleaning by careless handling.

8.3 If the test specimens are cut out 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, which remains stable under the conditions of the test, such as paint, wax or adhesive tape.

³⁾ See Bibliography; Reference [12].

9 Arrangement of the test specimens

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

9.2 The angle at which the surface of the test specimen is exposed in the cabinet is very important. The test 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 of 15° to 25°. In the case of irregular surfaces, for example, entire components, these limits shall be adhered to as closely as possible.

9.3 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 test specimens may be placed at different levels within the cabinet as long as the solution does not drip from test specimens or their supports at one level onto other test specimens placed below. However, for a new examination or for tests with a total duration exceeding 96 h, location permutation of test specimens is permitted.

9.4 The supports for the test specimens shall be made of inert non-metallic material. If it is necessary to suspend test specimens, the material used shall not be metallic but shall be synthetic fibre, cotton thread or other inert insulating material.

10 Operating conditions

10.1 Operating conditions are summarized in <u>Table 3</u>.

Test method item	Neutral salt spray (NSS)	Acetic acid salt spray (AASS)	Copper-accelerated acetic salt spray (CASS)
Temperature	35 °C ± 2 °C	35 °C ± 2 °C	50 °C ± 2 °C
Average collection rate for a horizontal collecting area of 80 cm ²	1,5 ml/h ± 0,5 ml/h		
Concentration of sodium chloride (collected solution)	50 g/l ± 5 g/l		
pH (collected solution)	6,5 to 7,2 3,1 to 3,3 3,1 to 3,3		3,1 to 3,3
The ± tolerances given are the allowable operational fluctuations, which are defined as the positive and negative deviations from the setting of the sensor at the operational control set point during equilibrium conditions. This does not mean that the set value may vary by plus/minus the amount indicated from the given value.			

Table 3 — Operating conditions

10.2 Check the collection rate and other test conditions in the test cabinet, filled to a similar extent as during the test. An empty or a completely filled cabinet behaves differently. After it has been confirmed that the test conditions are within a specified range, stop spraying the salt solution, fill the test cabinet with test specimens and start the test.

Evaporation of collected solution inside the cabinet can have an effect on the concentration and pH. Care should be taken to only measure solution that has not been subject to significant evaporation.

10.3 The solution collected in each of the collecting devices ($\underline{6.5}$) shall have a sodium chloride concentration and a pH value within the ranges given in <u>Table 3</u>.

The average rate of collection of solution in each device shall be measured over a minimum period of 24 h of continuous spraying. A daily checking of the collection rate during the operation of the cabinet is recommended.

10.4 The test solution which has been sprayed shall not be re-used.

10.5 During operation, the tank for the salt solution shall be covered by a lid to prevent dust or other contaminants from influencing the solution and to prevent the concentration of sodium chloride and the pH from fluctuating.

11 Duration of tests

11.1 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 and 1 008 h.

11.2 Interruptions of the test shall be minimized. 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.

The total opening time per day shall not exceed 1 h.

11.3 If the end-point of the test depends on the appearance of the first sign of corrosion, the test specimens shall be inspected frequently with the requirements of 11.2.

11.4 A periodic visual examination of specimens under test for a predetermined period may be carried out, 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.

12 Treatment of test specimens after test

12.1 General

How to treat test specimens after testing should be included in the test specification or material specification given by the customer. It shall be agreed with the test parties before starting the test.

12.2 Non-organic coated test specimens: metallic and/or inorganic coated

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 the test specimens gently in clean running tap water, at a temperature not exceeding 40 °C, and then to dry them immediately in a stream of air, at an overpressure not exceeding 200 kPa and at a distance of approximately 300 mm.

NOTE ISO 8407 can be used for treatment of the specimen after the test.

12.3 Organic coated test specimens

12.3.1 Scribed organic coated test specimens

Clean the surface of the organic coated test specimens under running tap water directly after removing the test specimens out of the salt spray cabinet. A soft sponge may be used to remove dirt and salt rests out of the scribed area but not to remove evaluable corrosion phenomena. Remove the delaminated area around the scribe by one of the following methods:

- a) using a knife. Carefully remove the loose coating using a knife blade held at an angle, positioning the blade at the interface between the coating and the substrate and forcing the coating away from the substrate.
- b) using an adhesive tape.

NOTE Removing the organic coating (paint coating) depends on the kind of coating (paint) and its behaviour in wet conditions. If agreed by the interested parties, let the test specimens dry in room atmosphere for 24 h and then treat them as described under a) and b).

12.3.2 Organic coated but not scribed test specimens

Coated but not scribed test specimens shall be cleaned under running tap water so that corrosion products and/or corrosion phenomena which have to be evaluated are not influenced by cleaning.

13 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 or corrosion defects (i.e. pits, cracks, blisters, rusting or creep from scratches in the case of organic coatings) to be assessed by methods described in ISO 8993 or ISO 10289 and, for organic coatings, in ISO 4628-1, ISO 4628-2, ISO 4628-3, ISO 4628-4, ISO 4628-5 and ISO 4628-8 (see <u>Annex D</u>);
- d) time elapsed 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 It is good engineering practice to define the appropriate criteria in the specification for a coating or a product to be tested.

14 Test report

14.1 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 test specimen tested and, when appropriate, the average result for a group of replicate test specimens. Photographic records of the tested test specimens may, if required, accompany the report.

14.2 The test report shall contain information about the test procedure. This information may vary according to the purpose of the test and the guidelines prescribed, but a general list of the details likely to be required is as follows:

- a) a reference to this document, i.e. ISO 9227, and the test performed (NSS, AASS or CASS);
- b) type and purity of salt and water used;
- c) description of the material or product tested;
- d) dimensions and shape of the test specimen, and 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) number of test specimens subjected to the test representing each material or product;

- method used to clean test specimens after the test with, where appropriate, an indication of the h) loss in mass resulting from the cleaning operation;
- i) angle at which the tested surfaces were inclined;
- frequency and number of test specimen location permutations if required; i)
- start and end date, duration of the test and results of any intermediate inspections; k)
- 1) properties of any reference specimens placed in the cabinet to check the stability of the operating conditions;
- m) test temperature;
- volume of the collected solution; n)
- pH of the test solution and the collected solution; 0)
- salt concentration or density of the collected solution; p)
- result of corrosion rate of reference specimens made of steel, or for both steel and zinc (mass q) loss, g/m^2);
- g th. any abnormality or incident occurring during the entire test procedure; r)
- intervals of inspection. s)

Annex A (informative)

Example schematic diagram of one possible design of spray cabinet with means for optional treating fog exhaust and drain



Figure A.1 — Schematic diagram of one possible design of spray cabinet (front view)



Кеу

- 1 test specimen
- 2 test specimen support
- 3 exhaust air treatment unit
- 4 air-outlet port
- 5 drain-treatment unit
- 6 salt tray
- 7 heating elements

Figure A.2 — Schematic diagram of one possible design of spray cabinet (side view)

Annex B

(informative)

Complementary method for evaluating cabinet corrosivity using zinc reference specimens

B.1 Reference specimens

As a complementary method for measuring cabinet corrosivity in accordance with this document, at least four reference specimens of zinc with an impurity level of less than 0,1 %, in mass fraction, may be used. The reference specimens should have dimensions of 50 mm \times 100 mm \times 1 mm.

Before testing, the reference specimens should be cleaned carefully with a hydrocarbon solvent in order to remove all evidence of dirt, oil, or other foreign matter liable to influence the result of the corrosion rate determination. After drying, the reference specimens should be weighed to the nearest 1 mg.

Protect one face of the reference specimens with a removable coating, for example, an adhesive plastic film.

B.2 Arrangement of the reference specimens

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

The support for the reference specimens should be made of, or coated with, inert materials such as plastic, and should be placed at the same level as the test specimens.

The recommended test duration for the NSS test is 48 h, for the AASS test 24 h and for the CASS test 24 h.

The cabinet should be verified during the testing of test specimens. If this is the case, great care shall be taken that the specimens do not affect each other. Otherwise, the cabinet shall be filled with substitute specimens to maintain the homogeneity of the cabinet. The verification procedure shall be performed using the same settings as for the test runs.

B.3 Determination of mass loss

Immediately after the end of the test, firstly remove the protective coating, then remove the corrosion products by repetitive cleaning as described in ISO 8407. For chemical cleaning of the zinc reference specimens, use a solution of saturated glycine, 250 g \pm 5 g of glycine, C₂H₅NO₂ (p.a.), per litre of deionized water.

The chemical cleaning procedure is preferably performed in repetitive immersions of 5 min. After each immersion step, thoroughly clean the reference specimen at ambient temperature by rinsing with water and by light brushing, then by rinsing with acetone or ethanol, followed by drying. Weigh the reference specimen to the nearest 1 mg and plot the mass versus the actual cleaning cycle as described in ISO 8407.

NOTE For efficient dissolution of the corrosion products during the immersion step, it is important that the solution be stirred continually. An ultrasonic bath might be preferred in order to increase the rate of dissolution.

From the plot of mass versus number of cleaning cycles, determine the true mass of the zinc reference specimen after removal of the corrosion products as described in ISO 8407. Subtract this number from the initial mass of the reference specimen prior to testing, and divide the resulting number by the area

of the exposed surface of the reference specimen to assess the metal mass loss per square metre of the reference specimen.

B.4 Satisfactory performance of cabinet

The cabinet has performed satisfactorily if the loss in mass of each zinc reference specimen is within the allowed ranges given in <u>Table B.1</u>.

Table B.1 — Allowed range of mass loss of the zinc reference specimens during verification of the corrosivity of the cabinet

Test method	Test duration h	Allowed range of mass loss of the zinc reference specimens g/m ²
NSS	48	50 ± 25
AASS	24	30 ± 15
CASS	24	50 ± 20

Annex C (normative)

Preparation of specimens with organic coatings for testing

C.1 Preparation and coating of specimens

Unless otherwise specified, each test specimen with organic coating shall be prepared in accordance with ISO 1514 and coated by the specified method with the product or system under test.

Unless otherwise specified, the back and edges of the specimen shall be coated with the coating system under test.

If the coating on the back or edges of the specimen differs from that of the coating product under test, it shall have a corrosion resistance greater than that of the coating product under test.

C.2 Drying and conditioning

Dry (or cure) and age (if applicable) each coated test specimen for the specified time under specified conditions and, unless otherwise specified, condition them at a temperature of 23 °C \pm 2 °C and a relative humidity of 50 % \pm 5 % for at least 16 h, with free circulation of air and without exposing them to direct sunlight. The test procedure shall then be carried out as soon as possible.

C.3 Thickness of coating

Determine the thickness, in micrometres, of the dried coating by one of the non-destructive procedures described in ISO 2808.

C.4 Preparation of scribe marks

If not otherwise agreed, all scribe marks shall be made in accordance with ISO 17872 and shall be at least 20 mm from each other and from any edge of the test specimen.

If specified, make a scribe mark through the coating to the substrate.

To apply the scribe mark, use a scribe instrument with a hard tip. Unless otherwise agreed, the scribe mark shall have either parallel sides or an upwards-broadening cross-section that shows a width of 0,2 mm to 1,0 mm at the metallic substrate.

One or two scribe marks may be applied. The scribe mark(s) shall be parallel to the longer edge of the test specimen, unless otherwise agreed.

The tool used to introduce scribe marks should give a uniform shape. Penknives shall not be used.

For aluminium specimens, two scribe marks shall be made which are perpendicular to, but do not intersect, each other. One scribe mark shall be parallel to the rolling direction and the other at right angles in accordance with ISO 4623-2:2016, Figure 1.

Annex D (normative)

Required supplementary information for testing test specimens with organic coatings

The items of supplementary information listed in this annex shall be supplied, as appropriate.

The information required should preferably be agreed between the interested parties and may be derived, in part or totally, from an international or national standard or other documents related to the system under test.

- a) Substrate material and surface preparation used (see <u>C.1</u>).
- b) Method of application of the test coating system to the substrate (see <u>C.1</u>).
- c) Duration and conditions of drying (or curing) and ageing (if applicable) of the test specimen before testing (see <u>C.2</u>).
- d) Thickness(es), in micrometres, of the dry coating(s), and method of measurement in accordance with ISO 2808, and whether it is a single coating or multicoat system (see <u>C.3</u>).
- e) Number and location of scribe marks made prior to exposure (see <u>C.4</u>).
- f) Duration of the test.
- g) Characteristics considered in evaluating the resistance of the test coating and method(s) of inspection used.

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