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Corrosion tests in artificial atmospheres - Salt spray tests (ISO 9227:2022)

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European foreword

This document (EN ISO 9227:2022) has been prepared by Technical Committee ISO/TC 156 "Corrosion of metals and alloys" in collaboration with Technical Committee CEN/TC 262 "Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys" the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2023, and conflicting national standards shall be withdrawn at the latest by May 2023.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 9227:2017.

Any feedback and questions on this document should be directed to the users' national standards body/national committee. A complete listing of these bodies can be found on the CEN website.

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Endorsement notice

The text of ISO 9227:2022 has been approved by CEN as EN ISO 9227:2022 without any modification.

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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.

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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, Corrosion of metals and alloys, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 9227:2017), which has been technically revised.

The main changes are as follows:

- the arrangement of test specimens has been added;
- the arrangement of collecting devices has been changed; examples of arrangement of collecting devices have been added as <u>Annex E</u>;
- DC04, DC05 and UNS G10080 have been added for steel reference specimens as an alternative of CR4grade steel, and interlaboratory comparison for reference specimens has been added as <u>Annex F</u>;
- the diluted acetic acid for preparing the test solution of AASS and CASS has been added;
- the allowed limit of copper concentration when the cabinet once used for CASS is re-used for NSS or AASS has been specified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 can 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.

When interpreting test results (e.g. minimum time until appearance defects or protection defects) for product quality control or acceptance specifications, it is important to recognize that the salt spray test can have a low level of reproducibility, especially with production parts tested in different laboratories.

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.

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 NSS test is particularly applicable to:

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

The 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 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

<u>ISO 4623-2:2016</u>, Paints and varnishes — Determination of resistance to filiform corrosion — Part 2: Aluminium substrates

ISO 8044, Corrosion of metals and alloys — Vocabulary

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

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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

reference specimen

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

Note 1 to entry: The reference material is the material with known test performance.

3.2

test specimen

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

3.3

substitute specimen

inert materials (such as plastic or glass) used for the substitute of a test specimen (3.2)

4 Principle

WARNING — This document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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

The AASS test is the test method in which an approximate 5 % sodium chloride solution acidified by the addition of acetic acid is atomized under a controlled environment.

The CASS test is the test method in which an approximate 5 % sodium chloride solution acidified by the addition acetic acid and with the addition of copper(II) chloride 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 to produce a concentration in a range between 45 g/l and 55 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 Anti-caking agents added to the sodium chloride 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 Preparation of each test solution with 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 test

Adjust the pH of the salt solution (see $\underline{5.1}$) so that the pH of the sprayed solution collected within the test cabinet ($\underline{6.2}$ and $\underline{6.5}$) is 6,5 to 7,2 at 25 °C ± 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 distilled or deionized water. Make any necessary corrections by adding hydrochloric acid, sodium hydroxide or sodium bicarbonate solution of analytical grade.

WARNING — Hydrochloric acid (CAS Registry Number^{®1)} 7647-01-0) solution is toxic, corrosive, irritating and very toxic to aquatic life. Refer to the safety data sheet for details. Handling of hydrochloric acid solution shall be restricted to skilled personnel or conducted under their control. Care shall be taken in the disposal of this solution.

WARNING — Sodium hydroxide (CAS 1310-73-2) solution is toxic, corrosive and irritating. Refer to the safety data sheet for details. Handling of sodium hydroxide solution shall be restricted to skilled personnel or conducted under their control. Care shall be taken in the disposal of this solution.

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 test

Add a sufficient amount of glacial acetic acid not less than 99,7 % of mass fraction or diluted acetic acid more than 10 % of mass fraction to the salt solution (see 5.1) to ensure that the pH of samples of sprayed solution collected in the test cabinet (6.2 and 6.5) is between 3,1 and 3,3 at 25 °C \pm 2 °C. Take the added volume of acetic acid into account when making up the initial sodium chloride solution. 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 distilled or deionized water. Make any necessary corrections by adding acetic acid, sodium hydroxide, or sodium bicarbonate of analytical grade.

WARNING — Glacial acetic acid (CAS 64-19-7) is a flammable liquid, toxic, corrosive and irritating. Refer to the safety data sheet for details. Handling of glacial acetic acid shall be restricted to skilled personnel or conducted under their control. Care shall be taken in the disposal of this solution.

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¹⁾ CAS Registry Number® is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.2.4 Copper-accelerated acetic acid salt spray test

Dissolve a sufficient mass of copper(II) chloride dihydrate ($CuCl_2 \cdot 2H_2O$) not less than 99,0 % of mass fraction in the salt solution (5.1) to produce a concentration of 0,26 g/l ± 0,02 g/l [equivalent to (0,205 ± 0,015) g of $CuCl_2$ per litre].

WARNING — Copper(II) chloride dihydrate (CAS 10125-13-0) is toxic, corrosive, irritating and very toxic to aquatic life. Refer to the safety data sheet for details. Handling of copper(II) chloride dihydrate shall be restricted to skilled personnel or conducted under their control. Care shall be taken in the disposal of this solution.

Adjust the pH using the procedures described in 5.2.3.

5.3 Filtration

If necessary, filter the solution before placing it in the reservoir of the apparatus, to remove any solid matter which can 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.

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 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 diluting salt solution prior to discharging it to the drainage system.

NOTE A schematic diagram of one possible design of spray cabinet is shown in \underline{Annex} A (see Figures A.1 and A.2).

6.3 Heater and temperature control

The test cabinet shall be maintained at the specified temperature (see 10.1) in the zone where the test specimens are placed by the appropriate system.

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 before introduction into the air humidifier to remove all traces of oil or solid matter, and the atomizing pressure shall be at an overpressure of 70 kPa to 170 kPa. The pressure is typically 98 kPa \pm 10 kPa but can vary depending on the type of cabinet and atomizer used.

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 and after the adiabatic expansion at the atomizer, 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. 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 kPa	Guiding values for the temperature, in °C, of the hot water in the saturation tower when performing the different salt spray test		
	NSS and AASS	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 made of inert material 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 atomizer 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 atomizer such that a continuous spray is achieved.

Distilled or deionized water with a conductivity not higher than 20 μ S/cm at 25 °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 cm^2 . The collecting devices shall be placed in the zone of the cabinet where the test specimens are placed. They shall be placed so that only mist, and not liquid falling from specimens or from parts of the cabinet, is collected.

The collecting devices shall be placed as follows.

a) During the test, two collecting devices should be placed at central points in the zone (see <u>Annex E</u> for an example).

- b) For calibration purposes, the collection rate of the cabinet shall be verified with at least six collecting devices, which are placed at the four corners and two central points of the zone (see Annex E for an example). This verification is done without test specimens in the cabinet, but preferably with substitute specimens (see also 10.2). It is recommended to perform it after installation, a move, modifications, adjustments or repair of the cabinet, after detecting nonconformities of the spray collection rate during running tests [see a)], and after idle periods longer than four weeks.
- c) If the collecting devices cannot be placed at four corners and/or at the central two points in the zone, they may be placed at another point by agreement between the interested parties. The number of collecting devices placed may also be changed according to the size of the cabinet by the agreement between the interested parties. In those cases, it shall be stated in the test report.

NOTE During permanent operation, a reasonable time period of the verification of the collection rate of the cabinet is generally considered to be three months.

When nonconforming spray collection rates are found during verification [see b)], the cabinet shall be adjusted or the test specimens shall not be placed in the nonconforming area.

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.

Additionally, it is recommended to check that the copper concentration in the collected solution is below the allowed limit of 2,5 mg/l (see 5.1), better below 0,5 mg/l, when the cabinet was previously used for CASS, but should now be used for AASS or NSS.

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 determine the corrosivity of the cabinet, reference specimens made of steel shall be used. It is necessary to verify the cabinet at regular intervals as described in 7.2 to 7.4.

NOTE 1 During permanent operation, a reasonable time period between two checks of the corrosivity of the cabinet is generally considered to be three months.

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>.

NOTE 2 The corrosivity of the cabinet verified with steel or high-purity zinc reference specimens via their mass loss does not guarantee reproducible times to the occurrence of certain corrosion products on coated specimens from industrial production.

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 surface and a matt finish (arithmetical mean deviation of the profile Ra = 0,8 μ m \pm 0,3 μ m). Cut these reference specimens from cold-rolled plates or strips. Alternatively to CR4-grade steel, the following steel grades

can be used, DC04 and DC05 according to EN 10130 and UNS G10080 according to SAE HS-1086 with reference to the results of the interlaboratory comparison (see Annex F).

NOTE 1 "Essentially faultless" means free from pores, marks, scratches and any light colouration.

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 a soft cloth, a 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.

NOTE 2 Cleaning with isopropanol can lead to a film of residues on the specimen surface.

WARNING — Most organic solvents are flammable liquids, toxic and irritating. Refer to the safety data sheet for details. Handling of organic solvents shall be restricted to skilled personnel or conducted under their control. Care shall be taken in the disposal of these solvents.

Determine the mass of the reference specimens to ± 1 mg. Protect one face of the reference specimens with a removable coating, e.g. 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 top of the collecting device should be in level with lower edge of the reference specimens or at the mean reference specimen exposure height.

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.

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 $\underline{ISO~8407}$. As one possibility of chemical cleaning, use a solution of diammonium hydrogen citrate [(NH₄)₂HC₆H₅O₇] (recognized analytical grade) in water with a concentration of 200 g/l 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 not performed satisfactorily if the mass loss of steel reference specimen is outside the allowed ranges given in Table 2.

Table 2 — Allowed range of mass loss of the steel reference specimens[14] 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 Annex B for the use of a 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 conforming to $\underline{ISO~1514}$, and of approximate dimensions $150~\text{mm} \times 100~\text{mm} \times 1~\text{mm}$. Annex C describes how test specimens with organic coatings shall be prepared for testing. Annex D 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 can attack the surface of the specimens.

Care shall be taken that test specimens are not re-contaminated 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.

9 Arrangement of the test specimens

- **9.1** The test specimens shall be placed in the zone fulfilling the operating condition of <u>Table 3</u>.
- **9.2** 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.
- **9.3** 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 (e.g. entire components), these limits shall be adhered to as closely as possible.
- **9.4** 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.
- **9.5** The supports for the test specimens shall be made of an inert non-metallic material or coated with an 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>.

Table 3 — Operating conditions

Test method item	NSS	AASS	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		o.	
pH (collected solution)	6,5 to 7,2	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.

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 (6.5) shall have a sodium chloride concentration and a pH value within the ranges given in Table 3.

The average rate of collection of solution in each device shall be measured over a minimum period of 24 h (including inspection time) 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, for changing the collecting devices, 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 and the cabinet should be opened not more than once per day, when the specified test duration exceeds 24 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 water, at a temperature not exceeding $40\,^{\circ}$ C, and then to dry them immediately in a stream of air, at an overpressure not exceeding $200\,^{\circ}$ kPa and at a distance of approximately $300\,^{\circ}$ mm.

NOTE <u>ISO 8407</u> 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 at a temperature not exceeding 40 °C 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:

- 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.

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;
- 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,

<u>ISO 8994</u> or <u>ISO 10289</u> and, for organic coatings, in <u>ISO 4628-1</u>, <u>ISO 4628-2</u>, <u>ISO 4628-3</u>, <u>ISO 4628-4</u>, <u>ISO 4628-8</u> (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;
- h) colorimetry.

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

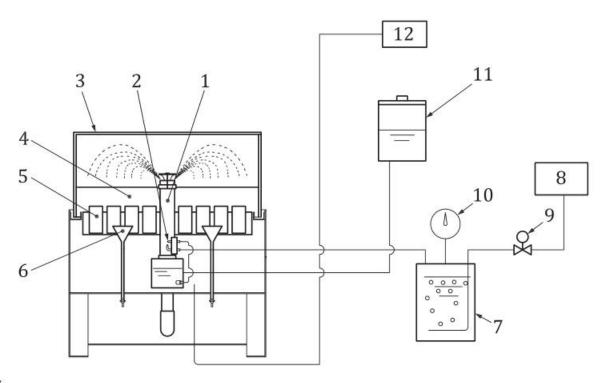
- **14.1** The test report shall indicate the outcome of the test according to the criteria for evaluation of results prescribed for the test. Report the result obtained for each test specimen tested and, when appropriate, the average result for a group of replicate test specimens. Photographic records 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 9227:2022, 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;
- h) 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) angle at which the tested surfaces were inclined;
- j) frequency and number of test specimen location permutations if required;
- k) start and end date, duration of the test and results of any intermediate inspections;
- properties of any reference specimens placed in the cabinet to check the stability of the operating conditions;
- m) test temperature;
- n) volume of the collected solution;
- pH of the test solution and the collected solution;
- p) salt concentration or density of the collected solution;

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- q) result of corrosion rate of reference specimens made of steel, or for both steel and zinc (mass loss, g/m^2);
- r) any deviations or incident occurring during the entire test procedure;
- s) intervals of inspection.

Annex A (informative)

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

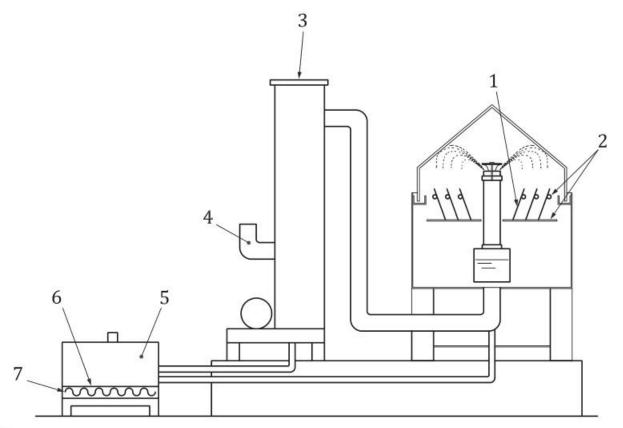


Key

- 1 dispersion tower
- 2 atomizer
- 3 cover
- 4 test cabinet
- 5 test specimen
- 6 collecting device

- 7 saturation tower
- 8 compressed air
- 9 solenoid valve
- 10 pressure gauge
- 11 solution tank
- 12 temperature controls

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



Key

- 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 $50 \text{ mm} \times 100 \text{ mm} \times 1 \text{ mm}$.

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, e.g. 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 saturated solution of glycine, $250 \text{ g} \pm 5 \text{ g}$ of glycine, $C_2H_5NO_2$ (p.a.), per litre of distilled or deionized water at $20 \, ^{\circ}\text{C}$ to $25 \, ^{\circ}\text{C}$.

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 can 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 not performed satisfactorily if the mass loss of zinc reference specimen is outside the allowed ranges given in <u>Table B.1</u>.

 ${\bf Table~B.1-Allowed~range~of~mass~loss~of~the~zinc~reference~specimens~during~verification} \\ {\bf of~the~corrosivity~of~the~cabinet}$

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 <u>ISO 1514</u> 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 <u>ISO 17872</u> 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.

Check the scribe to ensure there are no coating traces left in the scribe itself, and that the substrate is visible for the full length of the scratch.

Annex D

(informative)

Required supplementary information for testing test specimens with organic coatings

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

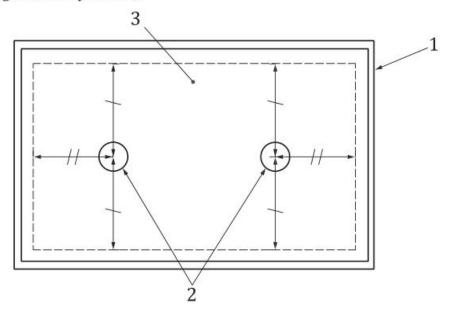
The information required should preferably be agreed between the interested parties and may be derived, in part or totally, from an international or national standard or other documents related to the system under test.

- a) Substrate material and surface preparation used (see C.1).
- b) Method of application of the test coating system to the substrate (see C.1).
- 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 <u>ISO 2808</u>, and whether it is a single coating or multicoat system (see <u>C.3</u>).
- e) Number, tool used, geometry and location of scribe marks made prior to exposure (see C.4).
- f) Duration of the test.
- g) Characteristics considered in evaluating the resistance of the test coating and method(s) of inspection used.

Annex E (informative)

Examples of arrangement of the collecting devices

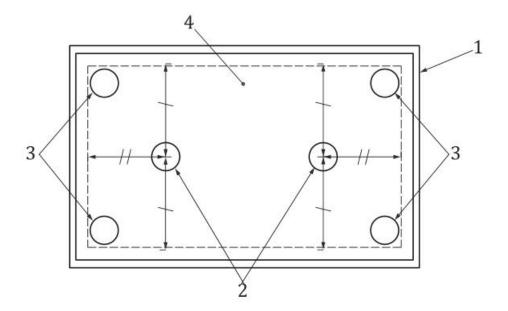
Figures E.1 and E.2 are examples of possible implementations of the arrangement of collecting devices. Any other arrangements may be used.



Key

- 1 test cabinet
- 2 collecting devices (at central two points)
- 3 zone of the cabinet where the test specimens are placed

Figure E.1 — Arrangement of collecting devices during the test, see 6.5 a)



Key

- 1 test cabinet
- 2 collecting devices (at central two points)
- 3 collecting devices (at four corners)
- 4 zone of the cabinet where the test specimens are placed

Figure E.2 — Arrangement of collecting devices for calibration purposes, see 6.5 b)

Annex F

(informative)

Interlaboratory comparison for reference specimens

F.1 General

The interlaboratory comparison was carried out to add the other steel reference specimens other than CR4 grade in ISO/TC156/WG7 from December, 2021 to February, 2022.

NOTE It was decided to perform the corrosivity qualification with NSS and CASS under the assumption that when a steel type reaches in both "extreme" tests similar mass losses as CR4, it can also be considered acceptable for AASS.

F.2 Reference specimens

The three candidates (DC04 and DC05 of EN 10130 and UNS G10080 of SAE HS-1086) were used. The CR4 of ISO 3574 was also used as reference. The dimension of each reference specimen is 150 mm \times 70 mm for CR4, DC04 and DC05, and 127 mm \times 76 mm for UNS G10080.

F.3 Participants of the interlaboratory comparison

The number of laboratories that participated in this interlaboratory comparison is as follows.

- NSS: 14 laboratories;
- CASS: 12 laboratories.

Each laboratory confirmed that the cabinet had been passed the corrosivity test according to ISO 9227:2017, Clause 7.

Each laboratory confirmed that the temperature and the collection rate in the cabinet used and pH and NaCl concentration/density of the collected solution were within the specifications of ISO 9227:2017, Table 3.

F.4 Procedure

F.4.1 Preparation

The 16 pieces of reference specimen were prepared for each cabinet according to ISO 9227:2017, 7.2. They were placed in the cabinet according to ISO 9227:2017, 7.3 except for the following deviations:

- a) four reference specimens (CR4, DC04, DC05 and UNS G10080) were placed in each quadrant;
- b) they were near each other, but not touching, obscuring, or otherwise influencing each other;
- whenever possible, CR4 was placed in the central position of each quadrant. DC04 and DC05 were placed on the left and right of CR4 or before and behind it respectively and UNS G10080 was also placed in close vicinity;
- d) a collecting device was placed near each group of the four reference specimens (according to ISO 9227:2017, 7.3).

The cabinet was filled with the substitute specimens as specified in ISO 9227:2017, 7.3.

F.4.2 Operation

The operation was in continuity 48 h for NSS and 24 h for CASS according to ISO 9227:2017, Table 2.

The collecting devices were removed immediately after the test. The collection rate, pH and NaCl concentration/density of the collected solution were evaluated separately for each quadrant.

After cleaning the reference specimens, the mass loss was determined individually according to $\underline{1SO~9227:2017}$, 7.4.

F.5 Test results

The test results are shown in Table F.1.

Table F.1 — Test results of NSS and CASS for all laboratories

Test	Steel reference specimen	Mean of mass loss	Allowed range of mass loss in ISO 9227:2017
		g/m ²	g/m ²
	CR4	64,2	
NCC	DC04	72,1	70 + 20
NSS	DC05	68,7	70 ± 20
	UNS G10080	69,7	
	CR4	52,5	
CACC	DC04	54,2	55.15
CASS	DC05	51,6	55 ± 15
	UNS G10080	49,9	

Bibliography

- [1] <u>ISO 3696</u>, Water for analytical laboratory use Specification and test methods
- [2] <u>ISO 4628-1</u>, 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
- [3] <u>ISO 4628-2</u>, 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
- [4] <u>ISO 4628-3</u>, 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
- [5] 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
- [6] <u>ISO 4628-5</u>, 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
- [7] <u>ISO 4628-8</u>, 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
- [8] ISO 8993, Anodizing of aluminium and its alloys Rating system for the evaluation of pitting corrosion Chart method
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- [10] <u>ISO 10289</u>, Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates Rating of test specimens and manufactured articles subjected to corrosion tests
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