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Corrosion of metals and alloys — Corrosion in artificial atmosphere — Accelerated corrosion test involving exposure under controlled conditions of humidity cycling and intermittent spraying of a salt solution

Corrosion des métaux et alliages — Corrosion en atmosphère artificielle — Essai de corrosion accélérée comprenant des expositions sous conditions contrôlées à des cycles d'humidité et à des vaporisations intermittentes de solution saline

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16701 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

As an alternative to the continuous salt spray test methods of ISO 9227[3], intermittent salt spray test methods may be used. The results of such tests provide a better correlation with the effects of exposure in environments where there is a significant influence of chloride ions, such as from a marine source or road de-icing salt.

Accelerated corrosion tests to simulate atmospheric corrosion in such environments should include cyclic exposure to the following conditions.

a) A wet phase, during which the test object is repeatedly subjected first to a spray of aqueous salt-containing solution then to a wet stand-by period during which residual wetness remains on the test object. This sequence provides a prolonged period of continuous exposure to wetness, extending over several hours.

b) A phase of controlled cyclic humidity conditions, where the test object is subjected to an environment which alternates between high humidity and comparative dryness.

These two phases should be cycled for an appropriate number of times.

The test method described in this International Standard conforms with these requirements, as follows.

In the first (wet) phase of exposure, the test objects are sprayed for 15 min with a 1 % (mass fraction) aqueous solution of sodium chloride acidified to pH 4,2, to simulate the rather acidic precipitation present in industrialized areas, followed by a 1 h 45 min period of wet stand-by. This sequence is repeated three times to give a total of 6 h exposure to wetness. The whole of this first phase of the test cycle is repeated twice a week. If spraying is carried out more frequently or a more concentrated solution of sodium chloride is used during this phase, phenomena may appear that are seldom experienced in the field, e.g. severe diffusion blocking by red rust or excessive dissolution of zinc.

The major part of the test cycle, however, consists of humidity cycling between the two levels of 95 % RH and 50 % RH at a constant temperature of 35 °C. To simulate the wet phase of humidity cycling, the humidity level has been set close to the condensation limit, but at a level at which test conditions can be satisfactorily controlled. Introducing 100 % humidity conditions, inevitably results in loss of control of the amount of salt deposited on a test object.

The test method described in this International Standard is mainly intended for comparative testing and the results obtained do not permit far-reaching conclusions on the corrosion resistance of the tested metallic material under the whole range of environmental conditions within which it may be used. Nevertheless, the method provides valuable information on the relative performance of materials exposed to salt-contaminated environments similar to those used in the test. See Annex A.

Corrosion of metals and alloys — Corrosion in artificial atmosphere — Accelerated corrosion test involving exposure under controlled conditions of humidity cycling and intermittent spraying of a salt solution

1   Scope

This International Standard defines an accelerated corrosion test method to be used in assessing the corrosion resistance of metals in environments where there is a significant influence of chloride ions, mainly as sodium chloride from a marine source or road de-icing salt.

This International Standard specifies the test apparatus and test procedure to be used in conducting the accelerated corrosion test to simulate, in a very controlled way, atmospheric corrosion conditions.

In this International Standard, the term "metal" includes metallic materials with or without corrosion protection.

The accelerated laboratory corrosion test applies to

⸺ metals and their alloys;

⸺ metallic coatings (anodic and cathodic);

⸺ chemical conversion coatings;

⸺ organic coatings on metals.

The method is especially suitable for comparative testing in the optimization of surface treatment systems.

2   Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 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 4628-4, *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 8407, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

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*

3   Reagent

A salt solution, prepared by dissolving a sufficient mass of sodium chloride in distilled or deionized water to a concentration of $10 \text{ g l}^{-1} \pm 1 \text{ g l}^{-1}$. The distilled or deionized water used shall have a conductivity not higher than 2 mS m^{-1} at $25 \text{ °C} \pm 2 \text{ °C}$.

The maximum permissible amounts of impurities in the sodium chloride are given in Table 1.

Table 1 — Maximum permissible amounts of impurities in the sodium chloride as calculated for dry salt

Impurity	Maximum mass fraction of impurity %	Note
Copper (calculated for dry salt)	0,001	Determined by atomic absorption spectro-photometry or other method with similar accuracy
Nickel (calculated for dry salt)	0,001	
Sodium iodide	0,1	
Total	0,5	

Check the pH of the salt solution by using potentiometric measurement at $25 \text{ °C} \pm 2 \text{ °C}$. Finely adjust the pH of the salt solution to a value of $4,2 \pm 0,1$ by standard addition of a diluted sulphuric acid solution (e.g. 1 ml of $0,05 \text{ N } H_2SO_4$ is added to 1 l of salt solution).

4   Apparatus

4.1   Climate chamber

The climate chamber shall be designed so that the following test conditions can be obtained, controlled and monitored during the test.

An instantaneous maximum deviation from set relative humidity value of ± 4 % in the range from 50 % to 95 % at 35 °C, which corresponds to a temperature accuracy requirement of $\pm 0,8$ °C at that temperature. For the mean value in relative humidity during a period of constant climatic conditions 7 h to 8 h an accuracy of ± 2 % shall apply, corresponding to temperature accuracy requirement of in this case $\pm 0,4$ °C.

NOTE   To meet the temperature and humidity accuracy requirements, the climate chamber should be equipped with means for efficient circulation of air to provide for small temperature and humidity variations in the chamber. Sufficient insulation of the chamber walls and lids are required in order to avoid excessive condensation on these surfaces.

The climate chamber shall also be designed so that the relative humidity may be changed linearly with respect to time from 95 % to 50 % within 2 h and back from 50 % to 95 % also within 2 h. Figure B.1 shows a suitable design of climate chamber.

The humidity and temperature levels of the climate chamber during a test cycle shall be continuously monitored or regularly checked so that it can be confirmed that the relative humidity versus time for a complete test cycle is within the specified ranges at 95 % RH and 50 % RH. For measurement of the relative humidity use a hygrometer designed for measurements at high humidity levels, e.g., a high-quality combined temperature and capacitance humidity sensor or a gold mirror dewpoint meter. For measurement of temperature preferably use Pt 100 sensors.

4.2 Spraying device

The spraying device for salt solution installed in the climate chamber shall be capable of producing a finely distributed uniform vertical downward flow of mist or small droplets falling on the test objects at a linear flow rate of 15 mm h^{-1} ± 5 mm h^{-1}.

If a graduated glass cylinder with a collecting area of 80 cm^2 is used to check whether this flow rate is within the specified range, the collection rate of salt solution shall be 120 ml h^{-1} ± 40 ml h^{-1}.

The device for salt spraying is preferably made of a number of nozzles mounted in series on a rail or tube. A spray pattern in the form of a fan, partly overlapping, is then obtained. The spraying device shall be made of, or lined with, materials resistant to corrosion by the salt solution and which do not influence the corrosivity of the sprayed salt solution. The use of molybdenum-alloyed stainless steel or plastic is recommended. Figure B.2 shows a suitable design of spraying device.

Salt solution that has already been sprayed shall not be re-used.

4.3 System for forced drying

The climate chamber shall be equipped with a system for forced air flow drying, as after spraying/wet stand-by all test objects should be dried from excessive macro wetness and climate control shall be made possible to regain within a reasonable time.

Forced drying is preferably arranged by supercooling and reheating an internal circulating flow. Alternatively, drying may be arranged by letting a forced flow of pre-heated ambient air ventilate the chamber. For a climate chamber of the volume 1 m^3 to 2 m^3 an air flow rate of 50 ls^{-1} to 100 ls^{-1} is recommended. The forced air flow shall not be pre-heated to such temperature levels that the maximum chamber temperature of 35 °C is exceeded.

NOTE   Pre-heating of the forced air flow to a temperature of 40 °C has been found suitable from practical experience.

5 Test objects

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

5.2 For each series of test objects, data records shall be kept and include the following information.

a) Specification of material to be tested; e.g., for surface-treated materials: type of base material, its pretreatment, type of coating, method of application and dry film thickness.

b) If the test specimen is subjected to intentional damage in the coating, the shape and the location of the damage should be described, as well as how the damage was achieved. The orientation of the damage during testing should also be specified.

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

c) Information on cleaning procedure to be used prior to testing.

d) Information on reference material or materials with which the test specimen is to be compared.

e) How the test object is to be examined and which properties are to be assessed, see e.g. Clause 7.

6   Procedure

6.1   Arrangement of the test objects

The test objects shall be placed in the cabinet on stands with their test surface facing upwards. The angle at which the surface of the test specimens is exposed in the cabinet is important. For flat test objects the angle at which the test surface is inclined shall preferably be $20° \pm 5°$ to the vertical. In the case of irregular surfaces, e.g. entire components, this angle shall be adhered to as closely as possible.

The stands with the test objects shall be placed on the same level of the climate chamber. The stands shall be made of inert non-metallic material, such as glass, plastic or suitably coated wood. If it is necessary to suspend the test object, the material used shall on no account be metallic but shall be synthetic fibre, cotton thread or other inert insulating material.

6.2   Exposure conditions of test cycle

6.2.1   Execute an exposure test in the climate chamber according to the following scheme of 12 h cycles. In the 1st cycle, the 8th cycle, the 15th cycle, and subsequently every seventh cycle, a cycle A with salt spraying as specified in 6.2.3 is used. In the other cycles, a cycle B as specified in 6.2.2 is used.

NOTE   From a practical point of view it may sometimes be preferable to perform the salt spraying on Mondays and Fridays. If the test is started on a Monday, accordingly, use cycle A in the 1st cycle, 9th cycle, 15th cycle, 21st cycle, 29th cycle, etc. In the other cycles, cycle B is used.

6.2.2   Cycle B is composed of the following steps, see also Figure 1:

— Step 1   Exposure at 35 °C and 95 % RH for 4 h.

— Step 2   Exposure at 35 °C to a linear reduction of relative humidity with time from 95 % RH to 50 % RH over a total period of 2 h.

— Step 3   Exposure at 35 °C and at 50 % RH for 4 h.

— Step 4   Exposure at 35 °C to a linear increase of relative humidity with time from 50 % RH to 95 % RH over a total period of 2 h.

6.2.3   Cycle A consists of the following steps, see also Figure 2:

— Step 5   Spraying of the test objects inside the climate chamber at 35 °C with salt solution at a linear downward flow rate of 15 mm h^{-1} for 15 min.

— Step 6   Exposure at 35 °C for 1 h 45 min with the relative humidity set point at 95 % to 99 % RH in such a way that the test objects remain wet.

Steps 5 and 6 are then repeated in sequence two more times to give a total period of wetness of 6 h.

— Step 7   Drying of the test objects, see 4.3, at a set (target) relative humidity of 50 % and at a temperature of 35 °C over a period of 4 h. The specified humidity level should be reached within 2 h, leaving the test objects and chamber interior without visible wetness.

— Step 4   Exposure at 35 °C to a linear increase of relative humidity with time from 50 % RH to 95 % RH over a total period of 2 h.
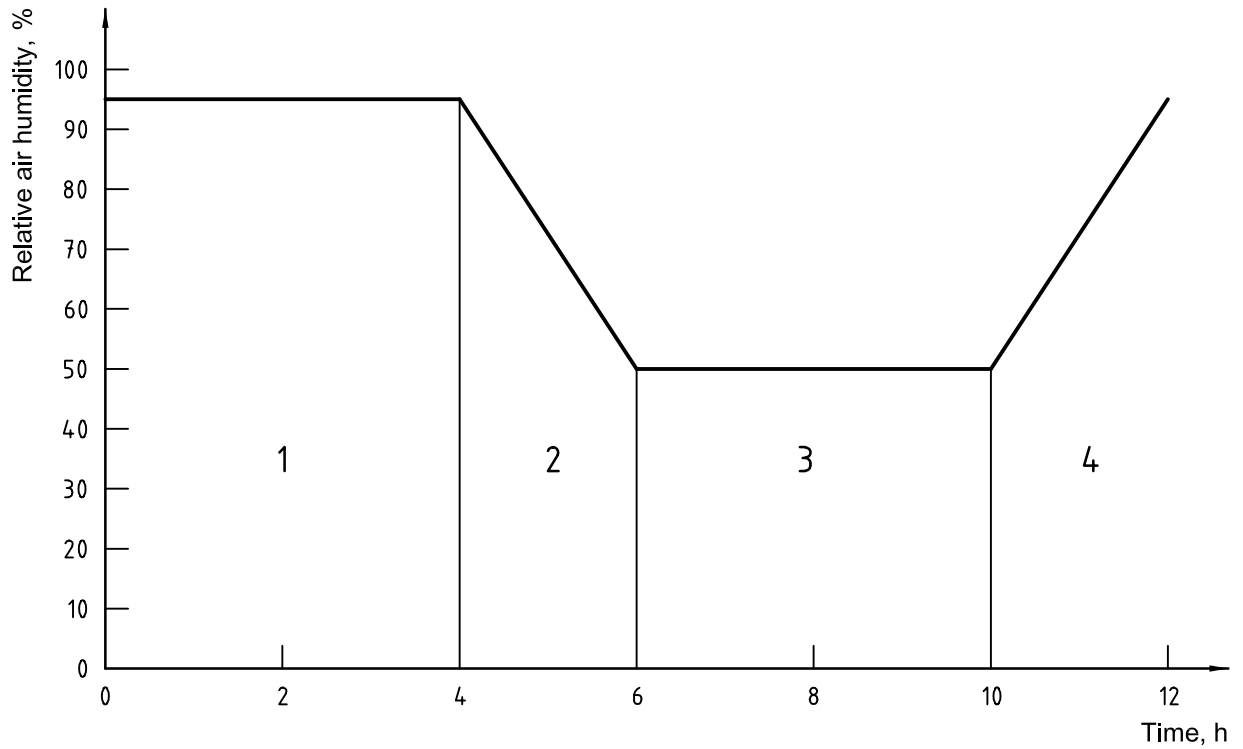
Figure 1 — Cycle B without spraying composed of the following sequence of steps: 1,2,3,4
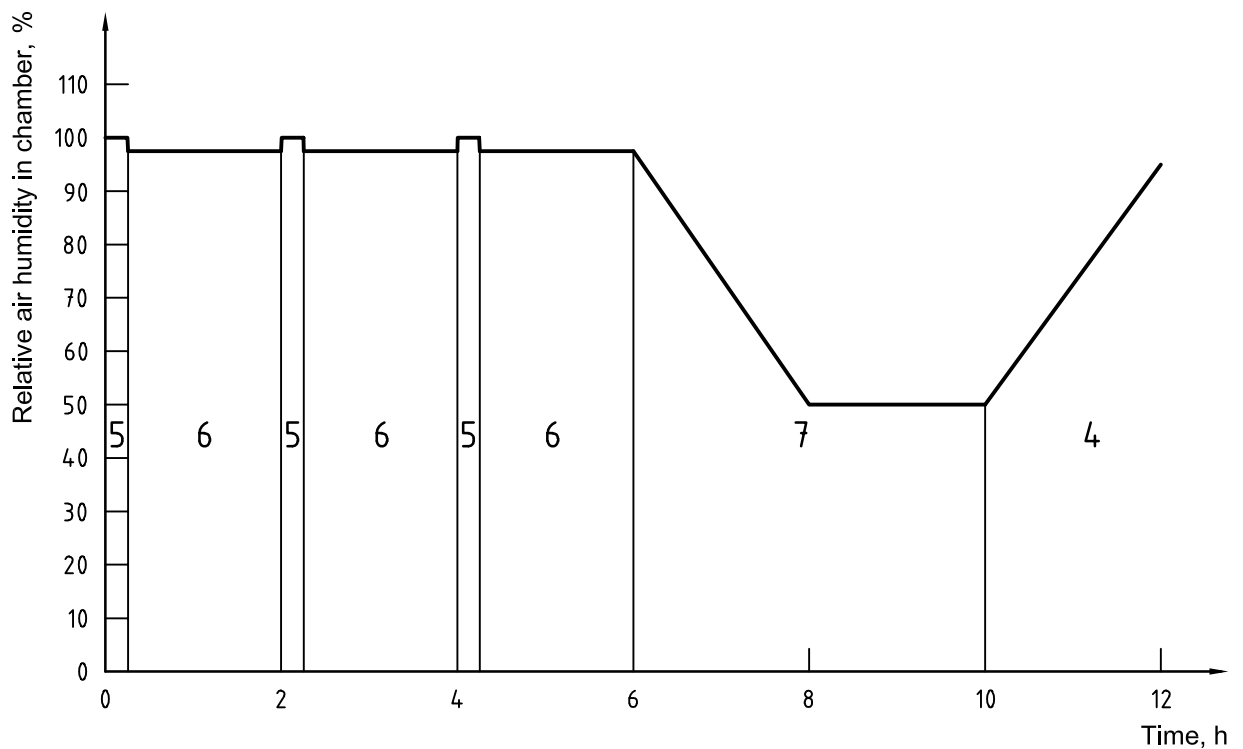

Figure 2 — Cycle A with spraying composed of the following sequence of steps: 5,6,5,6,5,6,7,4

NOTE 1   A simpler but less favourable alternative to spraying the test objects with salt solution inside the climate chamber is the use of manual alternate immersion of the test objects in salt solution outside the chamber. In this case it is recommended to replace cycle A with cycle C composed of the following steps:

— Step 5 a)   Remove the test objects from the climate chamber and immerse them for 15 min in the specified salt solution at a temperature of 35 °C. After emersion, manually spray the test objects with salt solution to restore droplets on the surface.

— Step 6 a)   After manual over-spraying let excessive fluid run off then return the test objects to the test chamber at 35 °C with the relative humidity set point at 95 % to 99 % for 1 h 45 min.

Steps 5 a) and 6 a) are then repeated in sequence two more times to give a total period of wetness of 6 h.

— Step 3   Exposure at 35 °C and at 50 % RH for 4 h.

— Step 4   Exposure at 35 °C to a linear increase of relative humidity with time from 50 % RH to 95 % RH over a total period of 2 h.

From a practical point of view it is further recommended that the manual immersion be performed on Mondays and Fridays so that cycle C with salt exposure is used: in the 1st cycle, 9th cycle, 15th cycle, 21st cycle, 29th cycle, etc. (the test is started on a Monday). In the other cycles, cycle B is used.

NOTE 2   Specimens of different metals should not be immersed in the same salt solution, in order to avoid contamination of dissolved ions of other metals (forming cathodes when reduced).

6.2.4   To check the reproducibility of test results, it is necessary to verify the corrosivity of the test at regular intervals.

NOTE   A suitable method for evaluation of the corrosivity using reference specimens is described in Annex C.

6.3   Duration of test

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

NOTE   Recommended periods of exposure for assessment of the corrosion resistance of different kinds of materials are given in Annex A.

6.4   Treatment of test objects after test

At the end of the test period, remove the test objects 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.

NOTE   This may be accomplished by dipping the test specimens gently in clean running water at a temperature not exceeding 40 °C and then drying them immediately in a stream of air at a pressure not exceeding 200 kPa at a distance of approximately 300 mm.

7   Evaluation of results

Many different criteria for the evaluation of the test results may be applied to meet particular requirements, e.g.

a)   appearance after the test;

b)   appearance after removing superficial corrosion products;

c)   number and distribution of corrosion defects, i.e., pits, cracks, blisters, etc.; these may be assessed by methods described in ISO 10289 and in ISO 4628-1, ISO 4628-2, ISO 4628-4 and ISO 4628-5;

d) the time elapsing before the appearance of the first signs of corrosion;

e) change in mass and evaluation of metallic mass loss in accordance with ISO 8407;

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.

8 Test report

The test report shall include following information:

a) reference to this International Standard, i.e., ISO 16701;

b) description of the test equipment;

c) the type, designation, dimensions and shape of the test object (material or product) and nature and area of the surface tested;

d) supplementary data in accordance with 5.2, such as preparation of the test object, including any cleaning treatment applied before testing and any protection given to edges;

e) the angle at which the tested surfaces were inclined during test;

f) the number of cycles or the duration of the test;

g) the frequency and intervals of inspection during testing;

h) the corrosivity of the test;

i) any deviations from the prescribed testing method;

j) test results after final evaluation of test objects, such as mass and thickness losses for uncoated specimens and blister width and peeling width for coated specimens, in accordance with Clause 7.

Annex A
(informative)

Recommended periods of testing

In general, a six-week test should be sufficient to rank any bare metal (alloy) or a metal protected by a thin conversion coating or a metallic, inorganic or organic coating. A twelve-week test is recommended for the ranking of high quality coating systems.

Table A1 illustrates how the recommended periods of testing according to this International Standard relate to two different kinds of field exposure test conditions in the case of cold rolled carbon steel and pure zinc (99,9 % mass fraction).

NOTE   If the more simple alternative to salt exposure as described in NOTE 1 in Clause 6.2.3 is used, the corrosivity of the test, or in other words, the metal thickness loss of cold rolled carbon steel and pure zinc after different periods of test is different from that given in Table A.1. Recommended periods of testing should in this case be based on what metal thickness losses are obtained for the reference specimens after different periods of testing.

Table A.1 — Comparison of metal losses due to corrosion in different tests

Material tested	Metal losses in thickness obtained after test µm		
	In accordance with this International Standard	In on-vehicle test after 2 y, vertical exposure, Gothenburg, see [5]	According to corrosivity class C5 in ISO 9223:1992 [2], first year data
Cold rolled carbon steel	115 to 130 (4 weeks of exposure) 180 to 220 (6 weeks of exposure) 315 to 385 (12 weeks of exposure)	80 to 150	80 to 200
Pure zinc	5 to 7 (4 weeks of exposure) 7 to 10 (6 weeks of exposure) 15 to 20 (12 weeks of exposure)	5 to 8	4,2 to 8,4

Annex B
(informative)

Suitable design of test apparatus with spraying device

## B.1 Climate chamber

See Figure B.1.
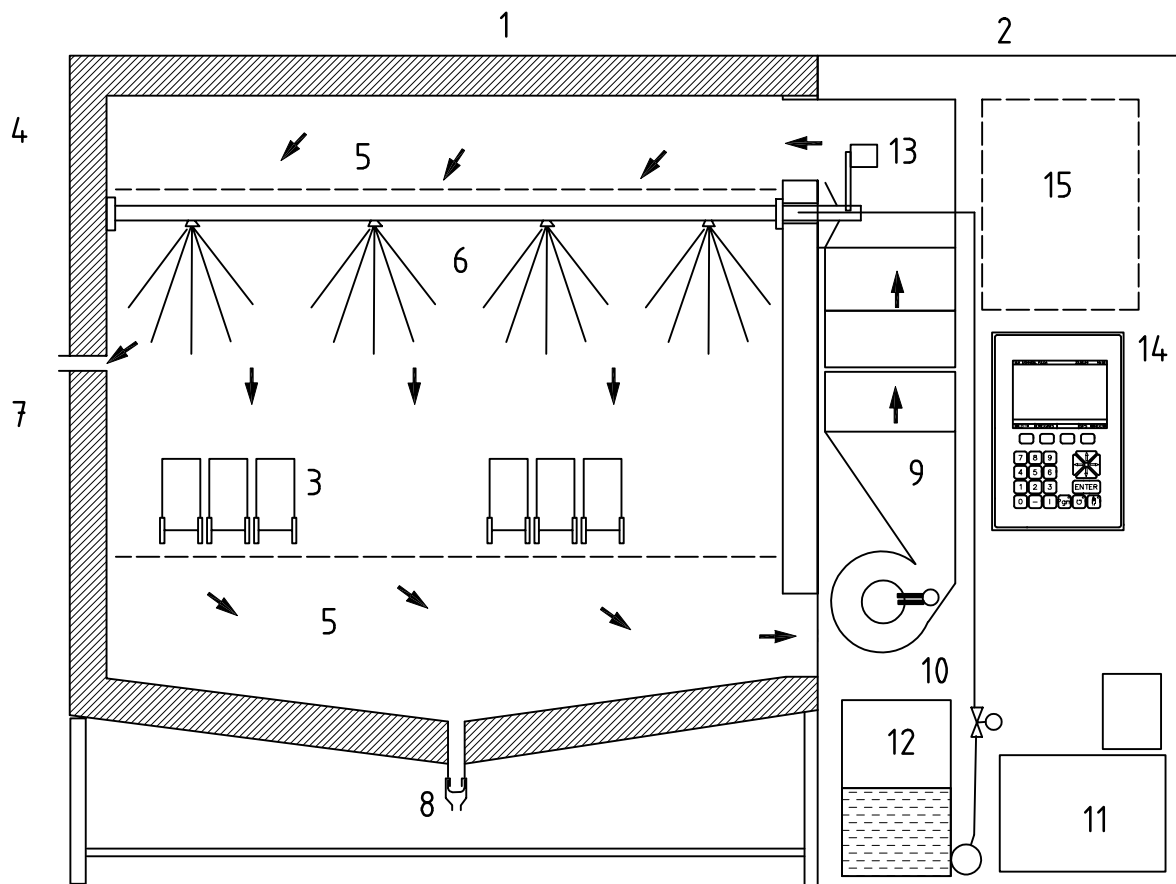
Key

1	test chamber	9	climatization unit (cooling/heating/humidification)
2	machinery unit	10	wet and dry Pt 100 sensors
3	test object area	11	cooling machine
4	well-insulated walls/lid	12	vessel with spray solution + pressurizing pump
5	air distribution grid plane	13	motor and link arms for swaying motion of precipitation rail
6	swaying rail with precipitation nozzles	14	control unit
7	air purge outlet	15	electronics and regulatory devices
8	precipitate sewer		

Figure B.1 — Climate chamber

B.2 Spray-rack design

See Figure B.2.
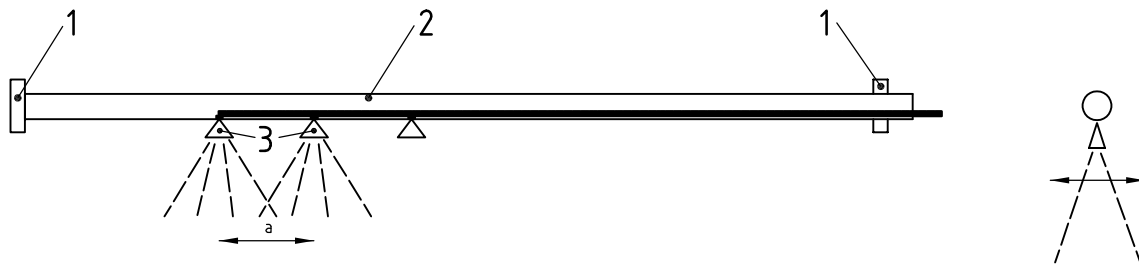
Key
1   bearing
2   tube/member
3   nozzles

a   0,5 m to 0,6 m.

Figure B.2 — Spray-rack design

Principle: Swaying tube or member supporting overlapping flat-spray nozzles.

Recommended nozzle type: Spraying Systems UniJet 800050VP. C/C mounting of nozzles on supporting tube 0,5 m to 0,6 m (approximately 1 m above test objects).

Swaying mode implemented by having supporting tube in bearings (free rotation, one side penetrating chamber wall) and rotating device with link-arms on outside of chamberwall.

Precipitation: 15 mm h^{-1} ± 5 mm h^{-1} throughout the whole rectangular test area (2 m^3, 4 nozzles in array).

Annex C
(informative)

Method for evaluation of corrosivity of test

C.1 Reference test specimen

For measurement of the corrosivity in the climate cabinet during the test in accordance with this International Standard, use four reference specimens each of

a) CR4 grade steel in accordance with ISO 3574, with a surface free from pores, slights, marks and scratches, and with a mat finish (arithmetical mean deviation of the profile Ra = 1,3 µm $\pm$ 0,4 µm) and

b) zinc with an impurity level of less than 0,1 % mass fraction.

The reference specimens should have dimensions of 50 mm $\times$ 100 mm $\times$ 1 mm.

Before testing, carefully clean the reference specimens using a hydrocarbon solvent, in order to remove all evidence of dirt, oil or other foreign matter capable of influencing the result of the corrosion rate determination. After drying, weigh the reference specimens to the nearest 0,1 mg.

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

C.2 Arrangement of the reference specimens

Position the four reference specimens of each material in four different quadrants in the climate cabinet, with the unprotected face upward, and at an angle of 20° $\pm$ 5° from the vertical.

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

C.3 Determination of mass loss

Immediately after the end of the test, remove the protective coating, then remove the corrosion products by repetitive cleaning as described in ISO 8407. Use the following chemical cleaning procedures:

a) for carbon steel a solution obtained by adding 20 g of antimony trioxide (Sb_2O_3)/1 000 g of hydrochloric acid and 50 g of tin(II) chloride ($SnCl_2$)/1 000 g of hydrochloric acid (ρ_{20} = 1,18 g/ml);

b) for zinc a solution of saturated glycine, 250 g $\pm$ 5 g glycine, $C_2H_5NO_2$ (p.a.)/1 000 ml deionized water.

The chemical cleaning procedure is, in both cases, preferably performed at room temperature, 20 °C to 25 °C, in repetitive steps of immersion of 5 min. After each immersion step, thoroughly clean the reference specimen at ambient temperature by rinsing with water followed by light brushing then rinsing with 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 continuously stirred. An ultrasonic bath may preferably be used for increasing the rate of dissolution.

From the plot of mass versus number of cleaning cycles, determine the true mass of the 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 area of the reference specimen to assess the metal mass loss per square metre of the reference specimen. Convert calculated mass loss per square metre into corrosion depth in µm by dividing by the density of the metal (density of carbon steel = 7,86 g/cm^3 and density of zinc = 7,14 g/cm^3).

C.4 Satisfactory operation of test apparatus

The test apparatus is considered to operate satisfactorily if the loss in mass of each specimen is within the range given in Annex A for the test duration used.

NOTE   If the simpler procedure for salt exposure is used, see NOTE in Annex A.

Bibliography

[1]   ISO 3574, *Cold-reduced carbon steel sheet of commercial and drawing qualities*

[2]   ISO 9223:1992, *Corrosion of metals and alloys — Corrosivity of atmospheres — Classification*

[3]   ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

[4]   STRÖM, M., STRÖM, G., OOIJ, W.J., SABATA, A., EDWARDS, R.A. and RAMAMURTHY, A.C., *A Statistically Designed Study of Atmospheric Corrosion Simulating Automotive Field Conditions Using a High Performance Climate Chamber — Status Report of Work in Progress*, SAE Technical Paper 912282, SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001 USA

[5]   STRÖM, M. and STRÖM, G., *A Statistically Designed Study of Atmospheric Corrosion Simulating Automotive Field Conditions Under Laboratory Conditions — Final Volvo Report on the AISI Cosmetic Corrosion Set of Materials*, SAE Technical Paper 932338, SAE International 400 Commonwealth Drive, Warrendale, PA 15096-0001 USA

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