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Corrosion of metals and alloys — Accelerated cyclic test with exposure to acidified salt spray, dry and wet conditions

Corrosion des métaux et alliages — Essais cycliques accélérés avec exposition au brouillard salin acidifié, en conditions «sèches» et en conditions «humides»

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Foreword

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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 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: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 16151:2005), which has been technically revised. The main technical changes are as follows:

- harmonization with ISO 9227;
- terms and definitions clause has been added.

Introduction

Corrosion of metallic materials, with or without corrosion protection, is influenced by many environmental factors, the importance of which may vary with the type of metallic material and with the type of environment. It is impossible, therefore, to design accelerated laboratory corrosion tests in such a way that all environmental factors influencing resistance to corrosion are taken into account. Laboratory tests are, therefore, designed to simulate the effects of the most important factors, which enhance the corrosion of metallic materials.

The accelerated corrosion-test methods described in this document are designed to simulate and enhance the environmental influence on a metallic material to outdoor climates, where exposure to acid rain and to salt-contaminated conditions occur and may promote corrosion. It has been prepared by reference to technical papers and reports (see the Bibliography).

The test methods involve cyclic exposure of test specimens to a mist of acidified-salt solution, to drying conditions, and to periods of high humidity. However, the methods are 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 in which they may be used. Nevertheless, the methods provide valuable information on the relative performance of materials exposed to salt and/or acid rain environments similar to those employed in the test.

Corrosion of metals and alloys — Accelerated cyclic test with exposure to acidified salt spray, dry and wet conditions

WARNING — This document may involve hazardous materials, operations and equipment. It does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices.

1 Scope

This document specifies two accelerated corrosion-test procedures, Methods A and B, for the comparative evaluation of metallic materials with or without permanent corrosion protection or temporary corrosion protection in outdoor salt and/or acid rain environments. It also specifies the apparatus used. The two tests involve cyclic exposure of the specimens to acidified salt spray, "dry" and "wet" conditions.

The particular advantages of the two tests over conventional accelerated tests, such as the neutral salt spray (NSS) test as specified in ISO 9227 lie in their better ability to reproduce the corrosion that occurs in outdoor salt and/or acid rain environments. They are also useful for evaluating cosmetic corrosion.

Method A is applicable to

— metals and their alloys,

— metallic coatings (cathodic),

— anodic oxide coatings, and

— organic coatings on metallic materials.

Method B is applicable to

— steel coated with anodic coatings, and

— steel coated with anodic coatings covered with conversion coatings.

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 4623-2:2016, *Paints and varnishes — Determination of resistance to filiform corrosion — Part 2: Aluminium substrates*

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

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:

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

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)

4 Test solution

4.1 General

Instructions for the preparation and use of the solutions used in Methods A and B are given in 4.2 and 4.3.

4.2 Method A

4.2.1 Neutral sodium chloride solution

A sufficient mass of sodium chloride shall be dissolved in distilled or deionized water, with a conductivity not higher than 20 µS/cm at 25 °C ± 2 °C, to produce a concentration of 50 g/l ± 5 g/l. The specific gravity range for a 50 g/l ± 5 g/l solution shall be 1,029 to 1,036 at 25 °C.

The sodium chloride shall not contain a mass fraction of the heavy metals of 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.

4.2.2 Preparation of the acidified-salt solution

The pH of the solution shall be adjusted to a value of 3,5 ± 0,1 at 25 °C ± 2 °C, by adding the following reagents to 10 l of the prepared neutral sodium chloride solution in 4.2.1 as follows:

- 12 ml of nitric acid (HNO_3, $\rho = 1{,}42$ g/ml);
- 17,3 ml of sulfuric acid (H_2SO_4, $\rho = 1{,}84$ g/ml);
- a sufficient quantity of 10 % mass fraction of sodium hydroxide (NaOH) solution, to adjust the pH of the solution to 3,5 ± 0,1 (about 300 ml will be required).

4.3 Method B

4.3.1 Preparation of the mixed salt solution

The reagent shown in Table 1 shall be dissolved in distilled or deionized water, with a conductivity not higher than 20 µS/cm at 25 °C ± 2 °C, to produce a stock solution with a concentration of 36,0 g/l ± 3,6 g/l. It shall be diluted by 1:6 to produce a mixed salt solution with a concentration of 6,0 g/l ± 0,6 g/l.

Table 1 — Composition and concentration of a stock solution for a mixed salt solution

Reagent	Concentration g/l
NaCl	24,53
$MgCl_2$	5,20
Na_2SO_4	4,09
$CaCl_2$	1,16
KCl	0,695
$NaHCO_3$	0,201
KBr	0,101
H_3BO_3	0,027
$SrCl_2$	0,025
NaF	0,003
WARNING — Handling of $SrCl_2$ and NaF can be hazardous and shall be restricted to skilled chemists or conducted under their control. NOTE The composition of the stock solution is the same as typical synthetic ocean water shown in ISO 11130.	

4.3.2 Preparation of the acidic solution

To prepare the acidic solution, 16,2 g of concentrated nitric acid (HNO_3, ρ = 1,40 g/ml, with a mass fraction of HNO_3 equal to 0,65) and 42,5 g of sulfuric acid (H_2SO_4, ρ = 1,84 g/ml, with a mass fraction of H_2SO_4 equal to 0,96) shall be dissolved in water and diluted to a total volume of 1 l to make 1 N acid solution with respect to nitric acid and sulfuric acid at an equivalent ratio of $[NO_3^-]/[SO_4^{2-}]$ of 0,4.

4.3.3 Preparation of the acidified-salt solution

The prepared acidic solution from 4.3.2 shall be added to the mixed salt solution from 4.3.1 to adjust the pH to 2,5 ± 0,1 at 25 °C ± 2 °C.

NOTE The relationship between the amount of mixed acidic solution from 4.3.2 and the pH of the acidified-salt solution is shown in Annex A. This solution, with pH value near to 2,5, would have a no-buffering action.

5 Apparatus

5.1 Component protection.

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

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.

5.2 Test cabinet.

The test cabinet shall be such that the conditions of homogeneity and distribution of the spray are met. Due to the limited capacity of test cabinets smaller than 0,4 m^3. The effect of the loading of the test 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 test cabinet so that it falls naturally down on them. The upper parts of the test cabinet shall be designed so that drops of sprayed solution formed on its surface do not fall on the specimens being tested.

The size and shape of the test cabinet shall be such that, during periods of exposure to salt mist spray, the rate of collection of solution in the test cabinet is within the limits specified in 8.2.

NOTE A schematic diagram of one possible design of test cabinet and associated apparatus for accelerated cyclic tests with exposure to acidified salt spray, "dry" and "wet" conditions is shown in Annex B.

5.3 Humidity and temperature control.

The temperature and humidity control system of the test cabinet shall be capable of achieving the test condition transition times listed in 8.1 when the test cabinet is filled with flat substitute specimens having a dimension of 150 mm × 70 mm × 1 mm.

5.4 Spraying device.

The device for spraying the acidified-salt solution shall comprise a supply of clean air, of controlled pressure and humidity, a reservoir containing the solution to be sprayed, and one or more atomizers.

The compressed air supplied to the atomizers shall be passed through a filter to remove all traces of oil or solid matter, and the atomizing pressure shall be at an overpressure of 70 kPa to 170 kPa. The pressure is typically 98 kPa ± 10 kPa but can vary depending on the type of test cabinet and atomizer used.

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

5.5 Air saturator.

In order to prevent evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer, by passage through a saturation tower containing hot water at a temperature higher than that of the test cabinet.

During periods of exposure to the acidified-salt solution mist, the appropriate temperature depends on the pressure used and on the type of atomizer nozzle and shall be adjusted so that the rate of collection of spray in the test cabinet, and the concentration of the collected spray, are kept within the specified limits (see 8.2). The level of water shall be maintained automatically to ensure adequate humidification.

5.6 Collecting devices.

At least two suitable collecting devices shall be available, consisting of clean funnels with a collecting area of approximately 80 cm^2, made of glass or other chemically inert material, with the stems inserted into graduated cylinders or other similar containers. The purpose of the collecting devices is to confirm that the collection rate is within specified limits (see 8.2). They shall be placed in the zone of the test cabinet where the test specimens are placed, at least one close to the spray inlet and one remote from the inlet and in such a way that only spray, and not the liquid falling from specimens or from parts of the test cabinet, is collected.

5.7 Air dryer.

A heating device and fan shall supply dried air of the specified humidity during "dry" periods (see 8.1).

5.8 Exhaust system.

The system by which air is exhausted from the test cabinet shall not be influenced by the back pressure of the atmosphere when the air is released at a point outside the building. It is preferred that the apparatus has a means to properly treat the fog prior to releasing it outside the building (see Annex B).

5.9 Drain system.

An appropriate system to treat the drain water shall be provided, so that the solution is not directly discharged into the drainage system (see Annex B).

6 Test specimens

6.1 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 with ISO 1514, and of approximate dimensions 150 mm × 100 mm × 1 mm. Annex D describes how test specimens with organic coatings shall be prepared for testing. Annex E gives supplementary information that shall be supplied, as appropriate, for testing test specimens with organic coatings.

6.2 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 re-contaminated after cleaning by careless handling.

6.3 If the test specimens are cut from a larger coated sample, 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.

7 Arrangement of the test specimen

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

7.2 If the test cabinet is not full of test specimens, it is recommended to fill it with inert flat substitute specimens of the same size as a reference specimen, in order to ensure uniformity of the spray. The material used shall be plastics, glass, or other inert insulating materials, which would not influence corrosion of the test specimens being tested.

7.3 The angle at which the surfaces of the test specimens are exposed in the test cabinet is very important. The specimen shall, in principle, be flat and placed in the test cabinet facing upwards at an angle as close as possible to 20° to the vertical. This angle shall, in all cases, be within limits 15° to 25°. In the case of irregular surfaces, for example entire components, these limits shall be adhered to as closely as possible.

7.4 The test specimens shall be arranged so that they do not come into contact with the test cabinet and so that the surfaces to be tested are exposed to free downward falling of spray. The specimens may be placed at different levels within the test cabinet, as long as the solution does not drip from specimens or their supports at one level onto specimens placed below them. However, for a new examination, or for tests with a total duration exceeding 96 h, location permutation of specimens is permitted. In this case, the number and frequency of the permutations are left to the operator and shall be indicated in the test report.