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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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Contents

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 document 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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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 third edition cancels and replaces the second edition (ISO 16701:2015), which has been technically revised.

The main changes are as follows:

- The test cycle has been revised with the same intended acceptance criteria to improve field correlation and reproducibility, when performed in various exposure chambers.
- An alternative cycle with alternating temperature in the climate cycling (constant dew point instead of constant temperature) is introduced. It is a modification of the so called ACT1 (originally Volvo Standard 423-0014), however within the same scope and intended acceptance criteria.
- Extensive requirements and guidelines on quality assurance of test equipment and control procedures have been incorporated, mainly as normative and informative annexes.

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

Atmospheric corrosion of metallic materials, with or without corrosion protection, is influenced by many environmental factors. Their corrosion impact varies with the type of exposed material system. It is therefore not possible to design a laboratory corrosion test in such a way that the complexity and variations of real, in-service conditions are fully represented. Acceleration (forced conditions) as such can also have a negative impact on the correlation-to-field performance. Nevertheless, tests with humidity cycling and only intermittent exposure to a salt solution provide a better correlation-to-field performance than tests using continuous salt spray.

This document was developed in the automotive context, where the major contributor to corrosion is the use of wintertime de-icing road salt in cool/cold temperate areas, most often as sodium chloride depositions that act in cyclic humidity conditions. The test procedure is moderately forced by humidity and salt. It is intended to be applicable for quality assurance of the metals and corrosion protections typically encountered in motor vehicles.[1]-[4] The method is also relevant in other areas of application, provided they have similar climatic conditions with an influence of sodium chloride depositions.[5][6]

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 document specifies an atmospheric accelerated test procedure in two closely related variants that contain intermittent salt exposure combined with dynamic humidity patterns: variant A at constant dew point and variant B at constant temperature. These variants are used in assessing the corrosion resistance of metals in environments where there is a significant influence of chloride ions, mainly as sodium chloride from, for example, winter road de-icing salt. 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 it can be used. Nevertheless, the method provides information on the relative corrosion resistance of a material exposed to a salt-contaminated environment with varying humidity conditions.

The two accelerated laboratory corrosion test variants are applicable to:

- metals and their alloys (ferrous and non-ferrous);
- metallic coatings;
- chemical conversion coatings;
- organic coating on metals;
- a combination of materials and coatings that include galvanic interactions and/or crevice conditions.

NOTE 1 If testing low-alloy stainless steels according to this document, especially austenitic grades, there is a risk of exaggerated pitting, which is not representative of most service environments.

NOTE 2 This document is not suitable for testing of wax and oil-based rust protection agents, due to the constantly elevated temperature condition of the test.

This document also specifies requirements on the test equipment and contains detailed procedures for quality control, including recommended instrumentation.

This document does not specify the dimensions of the tests specimens, the exposure period to be used for a particular product, or the interpretation of the results. Such details are provided in the appropriate product specifications.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
step
individual test climate operation, such as a linear climate ramp between two temperatures/humidities, constant conditions during a defined time period or spraying with salt solution

3.2
sequence
series of *steps* (3.1)

Note 1 to entry: Sequences can be handled as subroutines when programming the test.

3.3
sub-cycle
sequence (3.2) when run in a repeated mode

3.4
test cycle
full weekly (168 h) series of different *sub-cycles* (3.3), to be repeated for a stipulated number of weeks

3.5
test area
area within a test chamber (usually horizontal and designed as a structure open to air circulation) onto which test objects and their possible fixtures are supported

3.6
approved test area
part of the test area where the test conditions are fulfilled regarding climate and downfall of salt solution

3.7
test space
volume defined by the *test area* (3.5) times the permissible height to which the test object can extend without compromising the stipulated uniformity of the test conditions

3.8
approved test space
approved test area (3.6) times the height where the test conditions are fulfilled regarding climate and downfall of salt solution

3.9
tolerance span
permissible deviation ranges for the achieved momentaneous value of set temperature and relative humidity relative to the programmed set value at any time increment

3.10
macro wetness
droplets or an aqueous film visible to the eye, e.g. from spray application or by dew formation

4 Apparatus

4.1 General

The climate test chamber shall offer a design so that the requirements on the test conditions in Clause 5 can be obtained and controlled within the permissible range according to the procedures in Clause 7. The chamber test area and test space where these conditions are fulfilled are hence defined as the approved test area and approved test space, respectively. The test chamber design, applicable for manufacturing and at purchasing, shall be in accordance with Annex A.

4.2 Basic properties of the climate test chamber

For a fully automated procedure, the chamber shall be equipped with a spray device capable of producing a uniformly distributed spray of salt solution falling onto the test objects at a rate defined in 5.3. This can be performed by installing a swaying rail or tube equipped with flat-spraying nozzles as depicted in Annex B. Other technical solutions are permissible, provided they meet the downfall requirements.

4.3 Supporting requisites and accessories

The following requisites and accessories are necessary to perform the test method:

- Compressed, oil-free, clean air for humidification (if supplied by spray humidifiers), for cleaning salt solution from spray nozzles and as a source for the renewal of chamber air. The supplier of the equipment shall stipulate the specific requirements of the compressed air supply and may additionally include special requirements, such as that it should meet ISO 8573-1, class 4 or better.

- A low-conductivity water supply for humidification or evaporative spray humidifiers and for the preparation of salt solutions. The requirement on water quality for the equipment shall be set by the supplier of the equipment. From corrosion testing standpoint, it is sufficient to be below 20 µS/cm. If the humidification of the test chamber is supplied by high-pressure nozzle(s), the acceptable conductivity of the water can be as low as 5 µS/cm to secure the long-term performance of nozzles and heaters.

- A tank for preparation and storage of salt solution or, alternatively, a system for direct in-line mixing of water, saturated NaCl solution and, if applicable, diluted sulfuric acid to be contained within the stipulated concentration and tolerances.

- A conductivity meter with built-in temperature compensation for preparation and control of the salt solution.

- A standard pH-meter, with a glass-electrode for weak electrolytes, to be regularly calibrated at pH = 7 and pH = 4.

- Sodium chloride of min 99,9 % purity without sodium iodide addition, either as a vacuum salt supplied as grain salt or in accordance with the specification in ISO 9227.

- Sulfuric acid of reagent grade.
  WARNING — Sulfuric acid (CAS Registry Number®[1] 7664-93-9) solution causes severe skin burns and eye damage. Refer to safety data sheet for details. Handling of sulfuric acid solution shall be restricted to skilled personnel or conducted under their control.
  For accuracy, safety and convenience, it is recommended to use commercially available ampoules of diluted sulfuric acid (titration reagent) as a source for the preparation of the solution. One litre of a 0,5 M sulfuric acid solution prepared this way is sufficient for the acidification of 10 m^3 of ready salt solution. In the unlikely need to adjust up the pH of the ready salt solution, use diluted sodium hydroxide. For safety reasons, it is recommended to prepare the solution from commercially available titration ampoules. A 0,01 M solution is convenient.

- A device for independent monitoring of temperature and relative humidity (RH) shall be accessible. Annex C provides information on suitable devices. This instrument shall on stipulated intervals be used for independent monitoring and calibration of the climate in the test space of the exposure chamber in accordance with Annex D.

- Racks of inert material for support and aligned fixation of test object, which shall not hamper a free airflow around the test objects, nor collect standing wetness. Further requirements are found in Clause 6.

- Funnels, beakers/measuring glasses and a balance with at least 0,1 g accuracy for monitoring and calibration of salt solution downfall throughout the test plane of the exposure chamber.

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[1] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). 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.

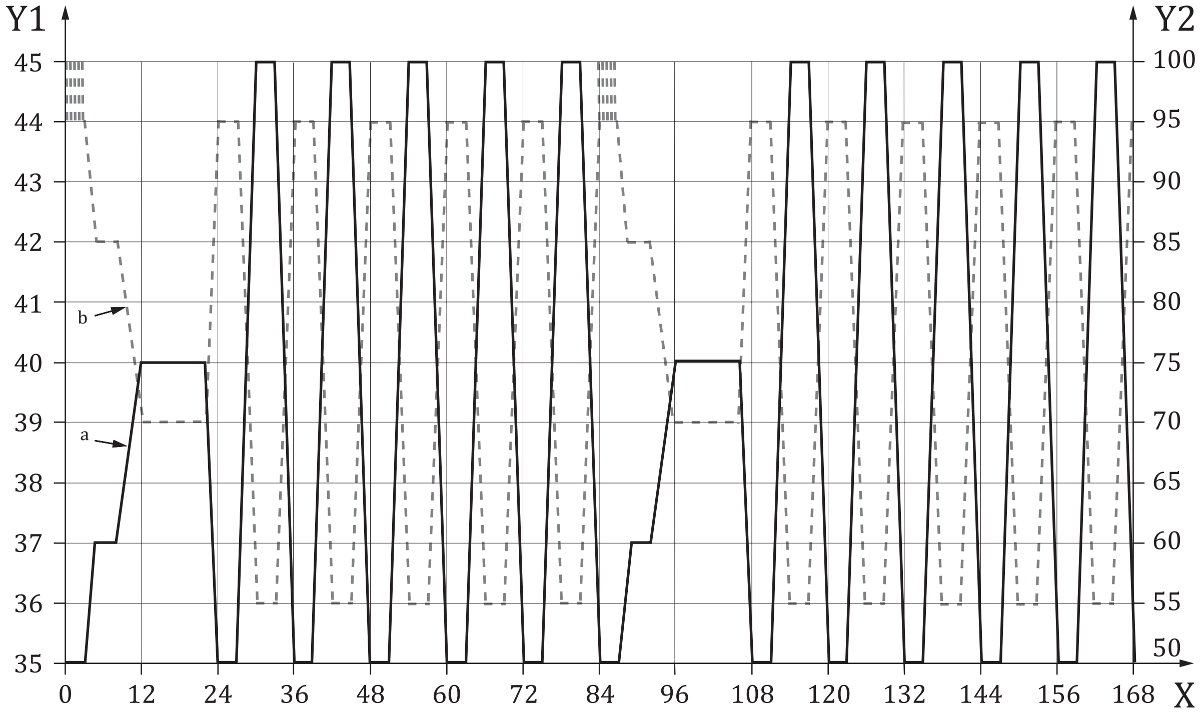
5 Test method

5.1 Test exposure conditions

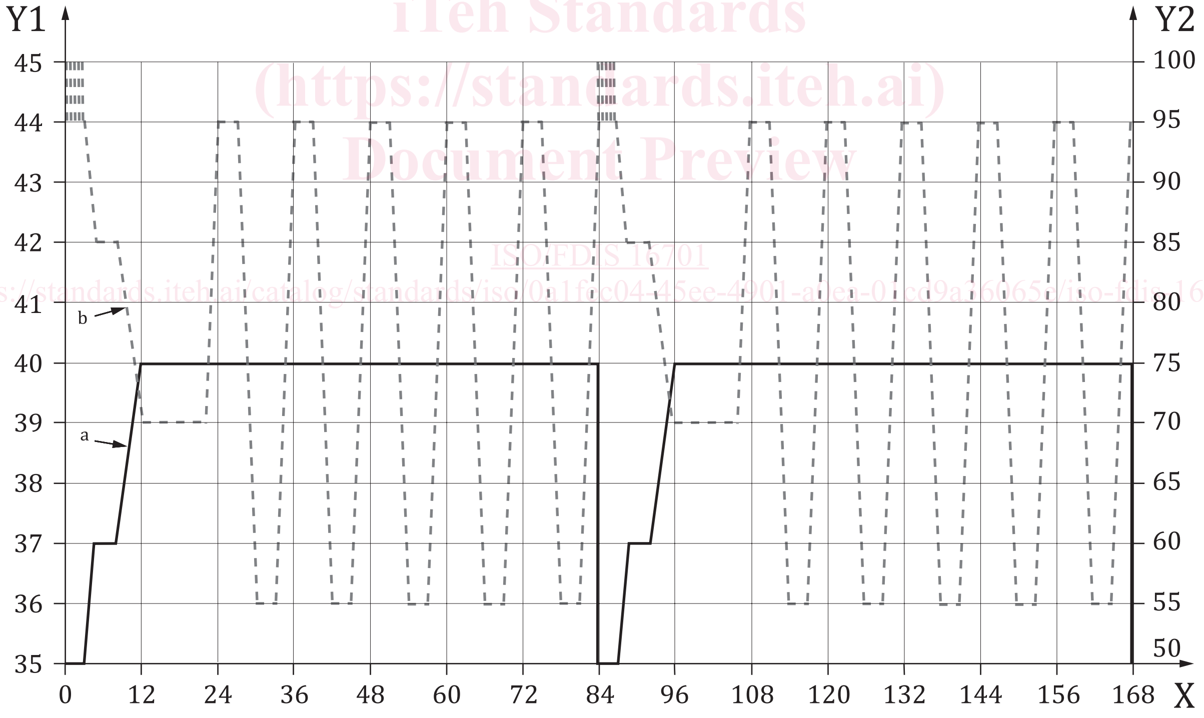
There are two variants of the test procedure: variant A and variant B. Figure 1 gives an overview of the weekly patterns of temperature and RH for the variants.

In variant A, the humidity cycling is run with a counter temperature cycling to receive approximately constant absolute humidity/dew point. This is in line with outdoor day and night conditions. Variant B runs with constant temperature during the humidity cycling and is recommended when heavy test objects are exposed.

Biweekly in both test variants, a 3-h wet period of intermittent spray with salt solution is followed by a controlled gradual drying-up for several hours (see Figure 2). This 24-h sequence is identical for variant A and B, and is followed by a series of repeated 12-h climate cycles, which are different for variants A and B (see Figure 3). The sequence of spraying followed by climate cycling is repeated once to reach a one-week test cycle. This cycle is then repeated for a stipulated number of weeks (typically six weeks) to form a complete atmospheric corrosion test. The background and rationales behind the two test variants and guidelines when to use which are found in Annex E. Table 1 and Table 2 list the detailed exposure conditions step by step for each test variant independently.



a) Weekly overview of test variant A

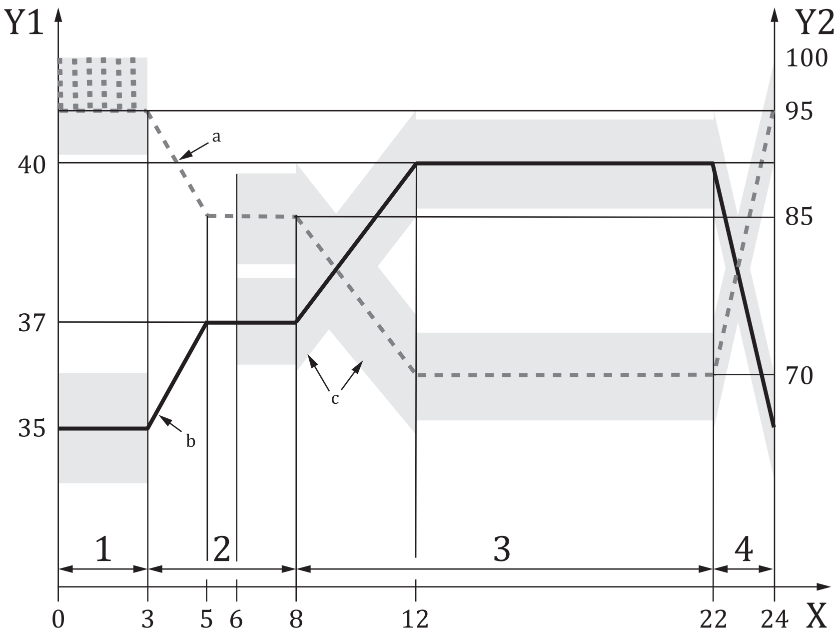


b) Weekly overview of test variant B

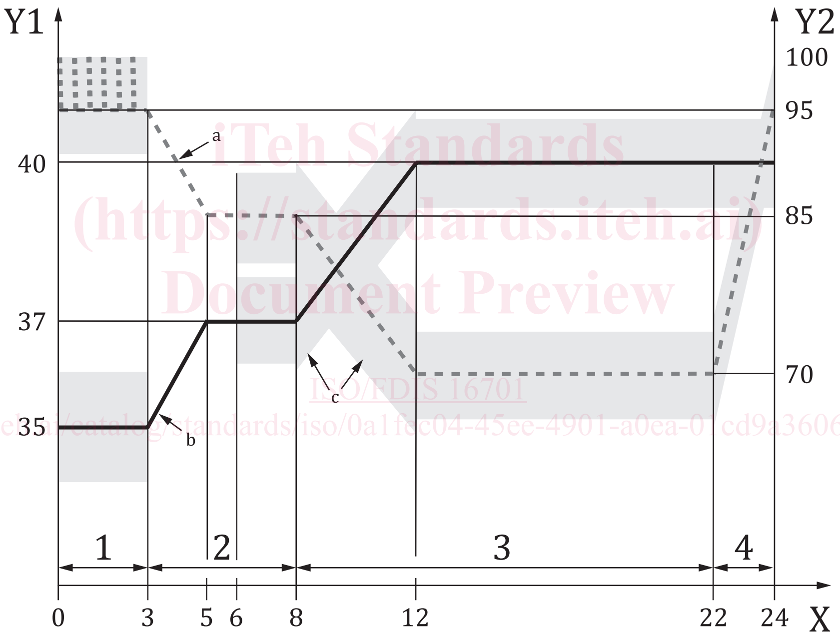
Key

X	elapsed time (h)	[a]	Temperature, expressed in solid line.
Y1	temperature (°C)	[b]	Relative humidity, expressed in dotted line.
Y2	relative humidity		

Figure 1 — Complete one-week cycles of the test variants A and B



a) Test variant A



b) Test variant B

Key

X	elapsed time (h)
Y1	exposure temperature (°C)
Y2	exposure relative humidity (% RH)
1	intermittent salt spray exposure
2	drying of corroded areas to 85 % RH
3	extended period of moderate drying
4	connection ramp to subsequent humidity cycling

[a]	Relative humidity, expressed in dotted line.
[b]	Temperature, expressed in solid line.
[c]	Allowed tolerance span in temperature and relative humidity, expressed in rasterized areas.

Figure 2 — 24-h precipitation and drying sequence for test variants A and B