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Corrosion tests in artificial atmospheres — Accelerated corrosion tests involving alternate exposure to corrosion-promoting gases, neutral salt-spray and drying

Essais de corrosion en atmosphères artificielles — Essais de corrosion accélérée par expositions alternées à des gaz oxydants ou au brouillard salin neutre et à un séchage

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Foreword

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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.

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

ISO 21207 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

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. Laboratory tests are designed to simulate the effects of the most important factors that enhance the corrosion of metallic materials.

The accelerated corrosion test methods described in this International Standard, methods A and B, are designed to simulate and enhance the environmental influence of exposure to an outdoor climate where salt-contaminated conditions and corrosion-promoting gases from an industrial or a traffic environment occur which may promote corrosion. Test method A simulates a moderately aggressive traffic environment while test method B simulates a more severe industrial or traffic environment.

Test method A involves exposure of the test objects to the following test cycle:

a) neutral salt spray testing (ISO 9227) for 2 h in a mist of a salt solution containing a mass fraction of 5 % of sodium chloride at 35 °C, followed by drying for 22 h in a standard laboratory climate;

b) exposure for 120 h in a test atmosphere containing a mixture of corrosion-promoting gases, volume fraction of NO_2 equal to $1{,}5 \times 10^{-6}$ and of SO_2 equal to $0{,}5 \times 10^{-6}$, at a relative humidity of 95 % and at a temperature of 25 °C, followed by drying for 24 h in a standard laboratory climate.

Test method B involves exposure of the test object to the following test cycle:

a) neutral salt spray testing (ISO 9227) for 2 h in a mist of a sodium chloride salt solution of mass fraction 5 % at 35 °C, followed by drying for 22 h in a standard laboratory climate;

b) exposure for 48 h in a test atmosphere containing a mixture of corrosion-promoting gases, volume fraction of NO_2 equal to 10×10^{-6} and of SO_2 equal to 5×10^{-6}, at a relative humidity of 95 % and at a temperature of 25 °C;

c) neutral salt spray testing (ISO 9227) for 2 h in a mist of a sodium chloride salt solution of mass fraction 5 % at 35 °C, followed by drying for 22 h in a standard laboratory climate;

d) exposure for 72 h in a test atmosphere containing a mixture of corrosion-promoting gases, volume fraction of NO_2 equal to 10×10^{-6} and of SO_2 equal to 5×10^{-6}, at a relative humidity of 95 % and at a temperature of 25 °C.

The results obtained do not permit far-reaching conclusions on the corrosion resistance of the tested product under the whole range of environmental conditions in which it may be used.

Corrosion tests in artificial atmospheres — Accelerated corrosion tests involving alternate exposure to corrosion-promoting gases, neutral salt-spray and drying

1   Scope

This International Standard defines two accelerated corrosion test methods to be used in assessing the corrosion resistance of products with metals in environments where there is a significant influence of chloride ions, mainly as sodium chloride from a marine source or by winter road de-icing salt, and of corrosion-promoting gases from industrial or traffic air pollution.

This International Standard specifies both the test apparatus and test procedures to be used in executing the accelerated corrosion tests.

The methods are especially suitable for assessing the corrosion resistance of sensitive products with metals, e.g. electronic components, used in traffic and industrial environments.

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 1337:1980, *Wrought coppers (having minimum copper contents of 99,85 %) — Chemical composition and forms of wrought products*

ISO 8407:1991, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

ISO 9227:1990, *Corrosion tests in artificial atmospheres — Salt spray tests*

ISO 10062:—[1]), *Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)*

ISO 10289:1999, *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   Apparatus and reagents

3.1   Exposure of test objects to mixtures of NO_2 and SO_2 at a relative humidity of 95 % and a temperature of 25 °C

The climatic cabinet with inner chamber and gas flow system shall comply with the requirements of ISO 10062.

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1)   To be published. (Revision of ISO 10062:1991)

The equipment used for testing shall be constructed so that

a) The inner chamber and gas flow system consist of inert materials, e.g. PTFE (polytetrafluoroethylene) or glass, to avoid or minimize adsorption of hostile gases on surfaces other than of that of the test panels.

b) The airflow and hostile gas injection system are designed to ensure uniform test conditions in the inner chamber or working space of cabinet.

NOTE   In the most common design of test equipment, the test atmosphere in the working space is obtained by continuously introducing the necessary quantity of the mixture of corrosion-promoting gases into a damp airflow to obtain the required concentration. The corrosion-promoting gases and conditioned air are mixed outside the cabinet. The conditioned air is taken from the outer chamber of the climatic cabinet. The airflow after injection of the corrosion-promoting gases is then mixed with a flow of re-circulated test atmosphere and the resulting gas flow admitted into the inner chamber or working space of the cabinet. Half of the flow of the test atmosphere through the inner chamber may be re-circulated.

To ensure uniform test conditions in the working space, the test atmosphere is normally supplied to the working space from the bottom and the outlet is placed at the top. Perforated plates are placed in front of the openings to assure uniform airflow through the working space.

c) Uniformity of temperature in the working space shall be better than ± 1 °C and uniformity of relative humidity better than ± 3 %. In terms of corrosivity, expressed as the corrosion rate of copper metal, the uniformity shall be not less than 5 %.

NOTE   The uniformity of the test conditions in the working space may be checked regularly by exposing a number of copper metal coupons, placed at different positions in the working space during testing. The differences in weight change of the metal coupons indicate if the uniformity of test conditions is within the specified range.

d) The tolerance of the damp air flow shall be within ± 1 °C of the specified temperature and the tolerance on relative humidity ± 3 %. The linear flow rate of air shall be in the range of 0,5 mm/s to 5 mm/s. The damp airflow shall be free of water droplets or aerosols.

NOTE   In the most common design of test equipment, the air is introduced to the outer chamber of the cabinet after filtration and purification by activated charcoal and a particulate filter. The nitrogen dioxide and the sulfur dioxide gases may be taken either from pressurized cylinders filled with 1 000 ppm (volume fraction 0,001 %) high-purity gases with a volume fraction of $1\ 000 \times 10^{-6}$ in high-purity nitrogen gas or from thermostat-controlled permeation tubes.

e) For exposure of test panels in the working space, specimen holders shall be used so that the test panels do not shield one another or disturb the uniformity of airflow across the chamber.

f) The temperature, relative humidity and concentration of sulfur dioxide and nitrogen dioxide in the airflow at the outlet of working space shall be monitored so that they reflect the true test conditions for the test objects.

3.2   Salt spray testing in accordance with ISO 9227

The apparatus and reagents to be used shall comply with the requirements of ISO 9227.

3.3   Drying in standard laboratory climate

For the test, a room with an appropriate system for control of its temperature at 23 °C $\pm$ 2 °C and its relative air humidity at 50 % $\pm$ 5 % shall be used.

4 Evaluation of the corrosivity of the tests

4.1 Reference test panels

For measurement of the corrosivity of the tests in accordance with this International Standard, four reference panels of metallic copper shall be used as specified for Cu-DHP in ISO 1337 (minimum 99,85 % mass fraction of Cu).

The reference specimens shall have dimensions of 50 mm × 50 mm × 1 mm.

Prior to exposure, grinding and polishing of the surfaces of the reference panels including edges shall be made by metallographic sample preparation equipment as follows:

a) the test panel shall be mounted on a flat specimen holder using double-sided tape;

b) the front surface of panel shall be ground on paper (220 grit) with a water lubricant until flat;

c) the front surface of panel and the edges of panel shall be polished for 5 minutes on cloth with 15 μm diamond suspension and an ethanol lubricant;

d) the panel shall be removed from the holder and steps 1 to 3 repeated but with the back side uppermost;

e) an identification number shall be stamped on the panel;

f) the panel shall be polished using 9 μm diamond suspension and tissue paper manually;

g) the panel shall be cleaned with ethanol and wiped with tissue paper manually;

h) the panel shall be cleaned in an ultrasonic bath with ethanol and then blown dry;

i) the panel shall be stored in a desiccator for a minimum period of 30 min;

j) the panel shall be weighed to the nearest 0,1 mg.

4.2 Arrangement of the reference panels during testing

During testing, the reference panels shall be handled in the same way as the test objects. During salt spray testing and exposure testing in the flow of the mixed corrosive gases, the four reference panels shall be positioned in the four different quadrants of the climate chamber and the salt spray cabinet.

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

4.3 Determination of mass loss after testing

After the end of the test, the corrosion products shall be removed from the reference panels by repetitive cleaning as described in ISO 8407.

For chemical cleaning, a solution with a mass fraction of 5 % of HSO_3NH_2 in distilled water shall be used. The chemical cleaning procedure is preferably performed in repetitive steps of immersion for 1 min at room temperature. After each immersion step, the reference panel shall be thoroughly cleaned at ambient temperature by immersion in water, then in ethanol, followed by drying. To avoid oxidation during chemical cleaning, the solutions should be saturated with nitrogen gas prior to and during the cleaning.

The reference panels shall be weighed to the nearest 0,1 mg and the mass versus the actual cleaning cycle shall be plotted as described in ISO 8407.

NOTE   For the efficient dissolution of the corrosion products during the immersion step, it is important that the solution be kept in a stirred state. Ultrasonic agitation is preferred for increasing the rate of dissolution.

From the plot of mass versus number of cleaning cycles, the true mass of the specimen after removal of the corrosion products shall be determined as described in ISO 8407. Subtract this number from the mass of the reference panel prior to testing and divide the resulting number by the area of the exposed surface of the reference panel to calculate the metal mass loss per square metre of the reference panel.

4.4   Satisfactory performance of test

The test has been performed satisfactorily if the loss in mass of each reference panel is within the intervals listed in Table 1.

Table 1 — Allowed ranges of mass loss of reference panel in the two test methods

Test method	Test duration	Allowed range of mass loss of reference panel g/m^2
A	2 weeks	$7,5 \pm 0,6$
	4 weeks	$14,5 \pm 1,5$
	6 weeks	$20,0 \pm 4,0$
B	1 week	$10,0 \pm 1,0$
	2 weeks	$25,0 \pm 2,5$
	3 weeks	$40,0 \pm 4,0$
	5 weeks	$70,0 \pm 7,0$

5   Test objects

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 so specified, these details shall be mutually agreed upon between the interested parties.

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

a)   Specification of the material or product to be tested.

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

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

d)   Information on the reference material or materials with which the test object will be compared.

e)   Test duration required.

f)   How the test object will be examined and which properties will be assessed.

6 Procedure

6.1 Test method A

6.1.1 Test cycle

The test objects shall be subjected to the following test cycle:

a) neutral salt spray testing (ISO 9227) for 2 h in a mist of 5 % (mass fraction) sodium chloride solution at 35 °C, followed by drying for 22 h in a standard laboratory climate, in accordance with 6.1.2

b) exposure for 120 h in a test atmosphere containing a mixture of corrosive gases, with a volume fraction of NO_2 equal to $1,5 \times 10^{-6}$ and of SO_2 equal to $0,5 \times 10^{-6}$, at a relative humidity of 95 % and at a temperature of 25 °C followed by drying for 24 h in standard laboratory climate, in accordance with 6.1.3.

6.1.2 Salt spray testing followed by drying (step a)

6.1.2.1   The test objects and the reference panels shall be placed in the salt spray cabinet and exposed to a mist of 5 % sodium chloride solution at 35 °C in accordance with ISO 9227. After 1 h of testing, the orientation of the reference panels shall be changed so that the surface facing upwards will become the surface facing downwards. After 2 h of testing, the test objects and the reference panels shall be removed from the cabinet and brought to a constant climate room without any rinsing.

6.1.2.2   The test objects and reference panels shall be stored in the constant climate room at 23 °C and 50 % RH, for 22 h before initiating step b) described in 6.1.3.

6.1.3 Exposure to corrosive gases followed by drying (step b)

6.1.3.1   The climatic cabinet shall be prepared for testing by first adjusting the temperature to 25 °C, the air humidity to 95 % RH and the air flow rate to a selected value between 0,5 mm/s and 5 mm/s. After stable conditions have been reached, the corrosive gas flows shall be adjusted so that the sulfur dioxide concentration in the inlet airflow to the working space shall be at a level of $0,5 \times 10^{-6}$ (volume fraction) and the nitrogen dioxide concentration in the inlet airflow to the working space shall be at a level of $1,5 \times 10^{-6}$ (volume fraction).

6.1.3.2   When stable conditions have been reached (also after this step), the door to cabinet shall be opened and the test objects and reference panels quickly placed in the working space. After a test period of not more than 6 h, the concentration of sulfur dioxide and nitrogen dioxide in the outlet airflow from the working space shall be not less than 80 % of that in the inlet airflow.

NOTE   If the concentration of the corrosive gases in the outlet airflow is lower, this probably means the total area of test objects in the cabinet is too large.

During the test, the exposure conditions shall be checked regularly and, if necessary, adjustments made to meet the specified levels.

6.1.3.3   After 120 h of testing, the test objects and the reference panels shall be removed from the working space of cabinet without subjecting them to any cleaning procedure, in accordance with 6.1.2. If the test is completed, the reference panels shall be placed in a constant climate room prior to weighing.

6.1.3.4   The test objects and reference panels shall be stored in the constant climate room at 23 °C and 50 % RH, for 24 h before initiating step a) described in 6.1.2.

6.1.4 Test duration

One test cycle corresponds to one week's exposure. The duration of the test shall be as designated by the specification covering the material or product being tested. When not specified, the period shall be agreed upon by the interested parties.

NOTE   Recommended periods of exposure for assessment of corrosion resistance are given in Annex A.

6.2 Test method B

6.2.1 Test cycle

The test objects shall be subjected to the following test cycle:

a) neutral salt spray testing (ISO 9227) for 2 h in a mist of 5 % (mass fraction) sodium chloride solution at 35 °C, followed by drying for 22 h in a standard laboratory climate, in accordance with 6.2.2;

b) exposure for 48 h in a test atmosphere containing a mixture of corrosive gases, with a volume fraction of NO_2 equal to 10×10^{-6} and of SO_2 equal to 5×10^{-6}, at a relative humidity of 95 % and at a temperature of 25 °C, in accordance with 6.2.3;

c) neutral salt spray testing (ISO 9227) for 2 h in a mist of 5 % (mass fraction) sodium chloride solution at 35 °C, followed by drying for 22 h in a standard laboratory climate, in accordance with 6.2.2;

d) exposure for 72 h in a test atmosphere containing a mixture of corrosive gases, with a volume fraction of NO_2 equal to 10×10^{-6} and of SO_2 equal to 5×10^{-6}, at a relative humidity of 95 % and at a temperature of 25 °C, in accordance with 6.2.3.

6.2.2 Salt spray testing followed by drying (step a or step c)

6.2.2.1   The test objects and the reference panels shall be placed in the salt spray cabinet and exposed to a mist of 5 % (mass fraction) sodium chloride solution at 35 °C in accordance with ISO 9227. After 1 h of testing, the orientation of the reference panels shall be changed so that the surface facing upwards will become the surface facing downwards. After 2 h of testing, the test objects and the reference panels shall be removed from the cabinet and brought to a constant climate room without any rinsing.

6.2.2.2   The test objects and reference panels shall be stored in the constant climate room at 23 °C and 50 % RH, for 22 h before initiating step b) or d) described in 6.2.3.

6.2.3 Exposure to corrosive gases (step b or step d)

6.2.3.1   The climatic cabinet shall be prepared for test by first adjusting the temperature to 25 °C, the air humidity to 95 % RH and the air flow rate to a selected value between 0,5 mm/s and 5 mm/s. After stable conditions have been reached, the corrosive gas flows shall be adjusted so that the volume fraction of sulfur dioxide in the inlet airflow to the working space shall be at a level of 5×10^{-6} and the volume fraction of nitrogen dioxide in the inlet airflow to the working space shall be at a level of 10×10^{-6}.

6.2.3.2   When stable conditions have been reached (also after this step), the door to the cabinet shall be opened and the test objects and reference panels quickly placed in the working space. After a test period of not more than 6 h, the concentration of sulfur dioxide and nitrogen dioxide in the outlet airflow from the working space shall be not less than 80 % of that in the inlet flow of air.

NOTE   If the concentration of the corrosive gases in the outlet airflow is lower, this probably means the total area of test objects in the cabinet is too large.

During the test, the exposure conditions shall be checked regularly and, if necessary, adjustments made to meet the specified levels.

6.2.3.3 After 48 h of testing if step b) is performed or after 72 h of test if step d) is performed, the test objects and the reference panels shall be removed from the working space of the cabinet without subjecting them to any cleaning procedure. Proceed to step c) if the test duration was 48 h or to step a) if the test duration was 72 h.

If the test is completed, the reference panels shall be placed in the constant climate room prior to weighing.

6.2.4 Test duration

One test cycle corresponds to one week of exposure. The duration of test shall be as designated by the specification covering the material or product being tested. When not specified, the period shall be agreed upon by the interested parties.

NOTE 1   Recommended periods of exposure for assessment of corrosion resistance are given in Annex A.

NOTE 2   It is advisable to start the test on Mondays in order to avoid work during weekends.

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)   number and distribution of corrosion defects, i.e. pits, cracks, blisters; (these may be assessed by methods described in ISO 10289);

c)   the time elapsed before the appearance of the first signs of corrosion;

d)   alteration revealed by micrographic examination;

e)   change in mechanical or electrical properties.

NOTE 1   It is good engineering practice to define the appropriate criteria in the specification for a product to be tested.

NOTE 2   The relative humidity should be $< 30 \%$ when measuring electrical properties, e.g. contact resistance, contact disturbance and insulation resistance. It is advisable to store the objects for a minimum of 48 h under these humidity conditions prior to measurements.

8   Test report

The test report shall include the following information:

a)   reference to this International Standard, i.e. ISO 21207;

b)   description of the test equipment;

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

d)   supplementary data in accordance with Clause 5, 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 test determined as described in Clause 4;

i)   any deviations from the prescribed testing method;

j)   test results after final evaluation of test objects.

Annex A
(informative)

Recommended test periods

Table A.1 shows recommended periods of test duration for simulating the corrosive load after certain years of exposure in a moderately aggressive traffic environment (test method A) and in a more severe traffic environment or industrial environment with salt contamination (test method B).

The recommended periods of test duration for test method A originate from a field study on copper corrosion in engine compartments of motor vehicles. The motor vehicles were used in road environments where de-icing salts contribute significantly to the atmospheric corrosivity, see references [2-4]. The recommended periods of test duration for test method B are based on data on copper corrosion at 39 different industrial sites in Europe and North America, see reference [5].

If the atmospheric corrosivity of the test under service conditions is measured and expressed in terms of the corrosion rate of a metal other than copper, the relation between years in a specified environment and corresponding test duration may be somewhat different from what is shown in Table A.1. In Table A.2 corresponding metal mass loss of metals other than copper in test method A and test method B is shown.

Table A.1 — Recommended periods of test duration

Test method	Type of environment	Years in specified environment	Assumed metal mass loss of copper defining corrosivity of specified environment g/m^2	Corresponding test duration
A	Moderately aggressive traffic (salt contaminated)	5	14,5	4 weeks
		8	20	6 weeks
B	Industrial (salt contaminated)	3	10	1 week
		8	25	2 weeks
		14	40	3 weeks
		27	70	5 weeks

Table A.2 — Corresponding metal mass loss of metals other than copper in test methods A and B

Test method	Test duration	Metal mass loss of copper g/m^2	Metal mass loss of zinc g/m^2	Metal mass loss of aluminium g/m^2	Metal mass loss of carbon steel g/m^2
A	4 weeks	14,5	40	4	—
B	3 weeks	40	90	7 to 10	580

Assuming 14 years of exposure, the data for test method B in Table A.2 can be used to estimate the corrosivity category for the service environment assumed for method B in Table A.1, with respect to all the different metals in Table A.2. From the average corrosion rates given for those metals in ISO 9224[1], corrosivity categories for copper, zinc and carbon steel can be estimated, which all fall within the lowest one third of the interval between corrosivity category 3 and corrosivity category 4. In the case of aluminium, the corrosivity category amounts to around 4.

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