## TECHNICAL REPORT

ISO/TR 16335

First edition 2013-09-01

#### Corrosion of metals and alloys — Corrosion tests in artificial atmospheres — Guidelines for selection of accelerated corrosion test for product qualification

Corrosion des métaux et alliages — Essais de corrosion en atmosphères artificielles — Lignes directrices pour un échantillon d'essais de corrosion accéléré pour la qualification du produit



#### ISO/TR 16335:2013(E)



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Published in Switzerland

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#### **Foreword**

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The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys.* 

#### Introduction

This document is a guideline for selection of suitable accelerated corrosion tests and is a survey of different internationally standardized test methods.

For that reason, this document is not suitable to be standardized but the document is greatly needed by the industry and test institutes.

The document has for that reason been prepared as a Technical Report.

In corrosion testing there has been a development from qualitative to more quantitative methods and the prerequisites for corrosion testing in product qualification are changing. Modern technologies for control and regulation of climatic test parameters are adopted in test equipment so that the reproducibility of tests increases. To make possible a better translation of laboratory test results into in-service performance, quantitative methods for characterization of corrosivity have been introduced during recent years. To evaluate the effect of corrosion attack on product functional performance, quantitative methods are adopted for assessing changes in the functional properties, as well as in the associated chemical changes resulting from corrosion of the materials of the component.

Field-site exposure testing was and still is the traditional way to verify the corrosion resistance of new materials and products, especially for testing new surface treatment systems or coatings for corrosion protection. Field test sites can be selected at places of high corrosivity as in marine or industrial areas. The field test sites therefore often represent worst cases of environments and as such the tests at those sites can be considered as accelerated tests. The degree of acceleration is, however, mostly moderate and it generally takes a long time to get an answer whether a tested material or product should be considered qualified with respect to its corrosion resistance.

For qualification of new materials and products with respect to corrosion resistance, therefore, accelerated corrosion tests generally need to be adopted during product design work. The higher the degree of acceleration of a corrosion test the more favourable the accelerated corrosion test will be in keeping the required testing time short. On the other hand, the higher the acceleration of the corrosion process needs to be during testing the harder it is to simulate properly the naturally occurring corrosion processes. This points at the main problem in designing meaningful accelerated corrosion tests for product qualification.

Large efforts have been made to develop accelerated corrosion tests for the purpose of product qualification. As a result of this work, a broad spectrum of methods now exists of which some are also available as International Standards. However, some of those tests are intended only for checking the comparative quality of a metallic material with or without corrosion protection, while others may even be useful for predicting or estimating the long-term performance of a product with metallic materials when exposed to corrosive stress representing in-service conditions.

To identify the most relevant method for one specific application requires knowledge that usually goes beyond what you can get from a single standard. This guideline therefore presents a framework for comparing existing accelerated corrosion tests so that the various aspects in the choice of best method and procedure can properly be taken into account.

# Corrosion of metals and alloys — Corrosion tests in artificial atmospheres — Guidelines for selection of accelerated corrosion test for product qualification

#### 1 Scope

This Technical Report is applicable for the selection of suitable accelerated atmospheric corrosion tests for qualification of products with metallic materials without or with permanent corrosion protection or temporary corrosion protection. The characteristics of a number of standardized accelerated corrosion tests are also given to serve as a guide in the preparation of test specifications.

In this Technical Report the following aspects are taken into account:

- Categories of accelerated atmospheric corrosion tests
- Recommended fields of application for the different kinds of tests and their suitability
- Corrosivity of tests and relative corrosion rates of standard metals
- Requirements for test equipment, criteria for reproducibility and correlation with in-service performance
- Recommended procedures for product qualification

The main purpose of this Technical Report is to present a framework for comparing the different accelerated corrosion test methods, which presently are available as International Standards. The suitability of a test method varies with the requirements set by the intended application of the product.

#### 2 Normative references

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

ISO 9223, Corrosion of metals and alloys — Corrosivity of atmospheres — Classification, determination and estimation

ISO 9224:2012, Corrosion of metals and alloys — Corrosivity of atmospheres — Guiding values for the corrosivity categories

ISO 9225, Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of environmental parameters affecting corrosivity of atmospheres

ISO 9226, Corrosion of metals and alloys — Corrosivity of atmospheres — Determination of corrosion rate of standard specimens for the evaluation of corrosivity

ISO 9227, Corrosion tests in artificial atmospheres — Salt spray tests

ISO 10062, Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)

ISO 11130, Corrosion of metals and alloys — Alternate immersion test in salt solution

ISO 11474, Corrosion of metals and alloys — Corrosion tests in artificial atmosphere — Accelerated outdoor test by intermittent spraying of a salt solution (Scab test)

ISO 11844-1, Corrosion of metals and alloys — Classification of low corrosivity of indoor atmospheres — Part 1: Determination and estimation of indoor corrosivity

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ISO 11997-1, Paints and varnishes — Determination of resistance to cyclic corrosion conditions — Part 1: Wet (salt fog)/dry/humidity

ISO 11997-2, Paints and varnishes — Determination of resistance to cyclic corrosion conditions — Part 2: Wet (salt fog)/dry/humidity/UV light

ISO 14993, Corrosion of metals and alloys — Accelerated testing involving cyclic exposure to salt mist, "dry" and "wet" conditions

ISO 16151, Corrosion of metals and alloys — Accelerated cyclic tests with exposure to acidified salt spray, "dry" and "wet" conditions

ISO 16701, 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

ISO 20340, Paints and varnishes — Performance requirements for protective paint systems for offshore and related structures

ISO 21207, Corrosion tests in artificial atmospheres — Accelerated corrosion tests involving alternate exposure to corrosion-promoting gases, neutral salt-spray and drying

IEC 60068-2-11, Environmental testing - Part 2: Tests. Test Ka: Salt mist

IEC 60068-2-30, Environmental testing - Part 2-30: Tests - Test Db: Damp heat, cyclic (12 h + 12 h cycle)

IEC 60068-2-52, Environmental testing - Part 2: Tests - Test Kb: Salt mist, cyclic (sodium, chloride solution)

IEC 60068-2-60, Environmental testing - Part 2: Tests - Test Ke: Flowing mixed gas corrosion test

IEC 60068-2-78, Environmental testing - Part 2-78: Tests - Test Cab: Damp heat, steady state

#### 3 Categories and characteristics of accelerated corrosion tests

The oldest and most wildly used method for laboratory accelerated corrosion testing is maybe the continuous neutral salt spray test (category A in  $\underline{\text{Table 1}}$ ). The continuous salt spray test is particularly useful for detecting discontinuities such as pores and other defects in certain metallic, anodic oxide and conversion coatings as well as in organic coatings. However, although used extensively for the purposes of qualification testing, results from continuous salt spray testing seldom correlate well with in-service performance.

Table 1 — Cate	gories of acc	elerated atmos	pheric corro	sion tests
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	Category of test	Examples of standards
A	Continuous salt spray tests	ISO 9227; IEC 60068-2-11
В	Tests with alternating immersion of test objects in a salt solution followed by drying or intermittent salt spraying and drying	ISO 11130;
С	Tests with cyclic variation of humidity (dry/wet) and including also steps of salt spraying	ISO 11474, ISO 14993; ISO 11997-1; ISO 11997-2; ISO 16151; ISO 16701, ISO 20340, IEC 60068-2-52
D	Tests with continuous exposure to atmospheres with low concentrations of corrosion promoting gases and at moderately high humidity	ISO 10062; IEC 60068-2-60
Е	Tests with continuous exposure to atmospheres with higher concentrations of corrosion promoting gases and at higher humidity including also steps of drying and short period of salt spraying	ISO 21207
F	High humidity tests	IEC 60068-2-78, IEC 60068-2-30, NT ELEC 025 (with condensation)
		See reference [1] in the Bibliography

One way to increase this ability is to introduce a step of drying after salt spray exposure (category B in Table 1). Even better, however is to combine salt spray exposure with humidity cycling between a high humidity level and a low humidity level (category C in Table 1) and, thus, introducing both wetting and drying in the corrosion test cycle.

Results from such tests turn out to correlate reasonably well with in-service performance at normal outdoor conditions. A number of cyclic accelerated corrosion tests based on this principle have been developed and standardized. The complexity of such tests, however, varies and so the requirements for test equipment. To get better control of the factors determining the rate of corrosion and relevance to in-service corrosion performance advanced systems have come into use.

Certain air pollutants as sulphur dioxide  $SO_2$ , nitrogen dioxide  $NO_2$ , hydrogen sulphide  $H_2S$ , and chlorine  $Cl_2$  present in air as trace substances promote corrosion of metals under high humidity conditions and need to be taken into consideration in the evaluation of corrosion resistance of products that are especially sensitive to corrosion failures such as electronic devices. High humidity exposure tests in the presence of such air pollutants are therefore frequently used in the qualification of electronic products with respect to corrosion resistance (category D in Table 1).

Corrosion effects may appear at air volume fractions of pollutants less than of 10-6. The conduct of air pollutant corrosion tests, therefore, requires very special kind of test equipment. Moreover mixtures of polluting gases are often used to simulate synergistic effects.

To assess corrosion resistance of certain products, tests combining intermittent salt spraying with exposure to corrosion promoting gases have also been introduced (category E in <a href="Table 1">Table 1</a>). Additional synergistic effects may be tested by such methods. The tests are also recommended for qualification of products designed for use in relative corrosive environments.

Sometimes tests involving exposure of test specimens to high humidity and to condensing water are considered as corrosion tests (category F in <u>Table 1</u>). Such test may produce corrosion effects on metallic parts of products if surface contaminants in the form of salts are present. Condensation tests are also used for the testing of organic coatings because they may induce damage caused by swelling and out-leakage of additives. For testing of electronic devices high humidity tests are used for control of air-tightness and in-leakage of water in the equipment. A special case of that is testing the corrosion protection capability of a semi permeable enclosure with electric device by initiating rapid cooling of the enclosure. This will cause the pumping of damp air into the enclosure and there result in condensation of water vapour if the cooling effect is sufficiently high.

## 4 Recommended fields of application for different kinds of tests and their suitability

During recent years methods for quantitative assessment and classification of atmospheric corrosivity have been developed and some of those exist also as International Standards. Atmospheric corrosivity for a specific location may either be estimated from meteorological data as described in ISO 9223 or assessed by measuring the corrosion rate of standard metal specimens at this location as described in ISO 9226.

The suitability of the different categories of corrosion tests for product qualification is given in <u>Table 2</u> for four different fields of applications and at varying corrosivity of an intended in-service environment in those applications.

The corrosivity categories C1 = very low corrosivity, C2 = low corrosivity, C3 = medium corrosivity, C4 = high corrosivity, C5 = very high corrosivity and CX = extreme corrosivity given in Table 2 are defined quantitatively in the standard ISO 9223. The severity classes C1 = mild, C2 = moderate, C3 = harsh, and C3 = severe appearing in Table 2 also are quantitatively defined in ISA S71.04 (Reference [2] in the Bibliography). Corrosivity classification for low corrosive atmospheres is also described in ISO 11844-1, which includes a comparison of ISO and ISA corrosivity categories.

For expressing the suitability of a specific category of corrosion test the following classes are used:

P = Preferred kind of method,

U = Useful for comparative testing of similar products, and

N = Not useful unless for quality control of the same product.

Table 2 — Suitability of corrosion test methods for different fields of application

Field of application		Suitability of different categories of corrosion tests					
Description	Corrosivity	A (constant salt spray)	B (alternate immersion)	C (humidity cycling with salt spray- ing)	D (air pollutant exposure)	E (air pollutant exposure, drying and salt spray)	<b>F</b> (condensation)
Marine construc-	Top site (C4-C5)	N	U	Р	-	p2)	-
tions	Splash (C5)	N	U	-	-	-	-
	Sub-sea <sup>1)</sup>	-	-	-	-	-	-
	Chassis (C4-C5)	N	U	Р	-	P2)	-
Automotive	Engine compartment (C2-C4)	N	U	Р	-	P <sup>2</sup> )	-
	Passenger compartment (C1)	-	-	-	P2)	-	P
	Open (C3-C5)	N	U	Р	-	P2)	-
Building construc- tions	Sheltered (C2-C4)	N	U	P	-	P <sup>2</sup> )	-
	Indoor (C1-C2)	-	-	-	P2)	-	Р
	Severe(GX)	fl3)	fl3)	Π3)	-	Р	P4)
Electric	Harsh (G3)	Π3)	fl3)	Π3)	-	Р	P4)
devices	Mild to Moderate (G1-G2)	-	-	-	P		Р

P = Preferred kind of method

U = Useful for comparative testing of similar products

N = Not useful unless for quality control of the same product

- 1) The total immersion test should be used.
- 2) Is the preferred kind of method for electric devices but is also of more general applicability.
- 3) For the testing of tightness.
- 4) Preferred kind of method when the effect of inner salt contaminants dominates.

General statements on the suitability of the different categories of tests for assessing the corrosion resistance of specific metallic materials are given in <u>Table 3</u> by making use of the same classes of suitability as used in <u>Table 2</u>.

Table 3 — Suitability of the different tests for assessing corrosion resistance of specific metallic materials with or without corrosion protection

	Suitability of different categories of corrosion tests							
Metallic material	A (constant salt spray)	B (alternate immersion)	C (humidity cycling with salt spraying)	<b>D</b> (air pollutant exposure)	E (air pollut- ant exposure, drying and salt spray)	<b>F</b> (condensation)		
Metals and alloys	N	U	Р	4)	P5)	7)		
Metals protected by cathodic coatings	U	U	Р	4)	P5)	7)		
Metals protected by anodic coatings	N	N	P1)	4)	P5)	7)		
Metals protected by conversion coatings on an anodic coating	N	U	Р	4)	P5)	7)		
Metals protected by organic coatings	N	U	p2), 3)	4)	P5)	p6), 7)		
Metals with temporary corrosion protection	-	-	-	-	-	P8)		

P = Preferred kind of method

U = Useful for comparative testing of similar products

N = Not useful unless for quality control of the same product

- 1) Consideration should be paid to the fact that some test methods enhance the corrosion of zinc relative to that of carbon steel, see <u>Table 5</u>.
- 2) Consideration should be paid to the fact that for some methods the drying times are too short and the salt load too high to avoid locking of paint under-creep corrosion for many coating systems. Crevice corrosion may be hampered by the same reason. In the selection of corrosion test method and specification of test severity consideration should be paid to the fact whether open air or crevice corrosion is the most critical.
- 3) Most methods are not capable to simulate all type of failure modes for painted aluminium.
- 4) Kind of test method mainly intended for testing of electric devices from mild to harsh corrosive environments.
- 5) Preferable kind of test method in connection with testing of electric devices but is also more generally applicable,
- 6) Condensation testing of value for checking wet adhesion of coatings.
- 7) Preferred kind of method for testing electric devices when the effect of inner salt contaminants dominates.
- 8) Temporary corrosion protection includes in this case surface treatment with waxes or other agents to protect metal surface from moisture.

In the table some considerations needed to be taken into account when selecting the most appropriate test method within a certain category of corrosion tests are also stated.

#### 5 Corrosivity of tests and relative corrosion rates of standard metals

The use of standard metal specimen exposure to assess corrosivity or corrosion load should preferably be adopted for characterizing the corrosive stress in a specific accelerated corrosion test. Data on corrosivity with respect to corrosion of standard metal specimens are available for many standard

tests and such data can be used to compare different tests. To illustrate how the corrosive stress varies between some standard tests, estimated mean testing times to reach a metallic mass loss of carbon steel of 670 g m $^{-2}$ , corresponding to approximately 5 years of outdoor exposure in corrosivity category C3 mean, according to ISO 9224:2012 are presented in <u>Table 4</u>. Data on the corrosivity of the different tests are found in the respective standards but are also available in <u>Annex A</u> of this Technical Report.

Table 4 — Testing time to reach a metallic mass loss of carbon steel of 670 g m<sup>-2</sup>, corresponding to approximately 5 years of outdoor exposure in corrosivity category C3 mean according to ISO 9224:2012

Test method	Mean testing time <sup>1)</sup> to reach a metallic mass loss of carbon steel = 670 g/m <sup>2</sup> due to corrosion (days)
Outdoor exposure under mean C3 atmospheric corrosivity conditions according to ISO 9224	1780
Outdoor exposure under mean C4 atmospheric corrosivity conditions according to ISO 9224	622
Outdoor exposure under mean C5 atmospheric corrosivity conditions according to ISO 9224	143
Outdoor exposure under mean CX atmospheric corrosivity conditions according to ISO 9224	15
ISO 9227	19
ISO 14993	7
ISO 16151A	12
ISO 16151B	15
ISO 16701	19
ISO 21207B	24
ISO 11997-1	32
ISO 11474	120 2)

<sup>1)</sup> Mean testing times have been estimated from metallic mass loss data found in the respective standards and it has further been assumed that metallic mass loss versus exposure time for the accelerated tests is linear

The corrosion rate of one standard metal in relation to the corrosion rate of another standard metal in an accelerated corrosion test should be considered in the choice of the most suitable test for a given application. The relative corrosion rate of the two standard metals in the test compared to the relative corrosion rate of the same metals under in-service conditions is a measure on how well the test reproduces in-service corrosion behaviour. To illustrate this, corrosion load data for carbon steel and for zinc in some standard accelerated corrosion tests are shown in Table 5. In this table estimated allowed range of metallic mass loss of carbon steel and of zinc with reference to a mean metallic mass loss of carbon steel equal to  $670 \text{ g/m}^2$  are shown in comparison with data from outdoor exposure during 5 years in atmospheric corrosivity category C3 according to ISO 9224:2012.

<sup>2)</sup> Data representative for testing during winter season at SP Technical Research Institute of Sweden

Table 5 — Allowed range of metallic mass loss of carbon steel and of zinc with reference to a
mean metallic mass loss of carbon steel equal to 670 g/m <sup>2</sup>

Test method/exposure time <sup>1)</sup>	Metallic mass loss of carbon steel (g/m²)	Metallic mass loss of zinc $(g/m^2)$	
Atmospheric corrosivity category C3 for 5 years	450 - 900	18 - 55	
ISO 9227 for 19 days	480 - 860	240 - 710	
ISO 14993 for 7 days	530 - 800	100 - 170	
ISO 16151 A for 12 days	460 - 880	140 - 250	
ISO 16151 B for 15 days	470 - 870	15 - 50	
ISO 16701 for 19 days	630 - 710	25 - 35	
ISO 21207 B for 24 days	670 2)	104 2)	
ISO 11997-1 for 32 days	670 2)	241 2)	
ISO 11474 for 4 months	670 2)	63 2)	

- 1) Mean testing times have been estimated from metallic mass loss data found in the respective standards and it has further been assumed that metallic mass loss versus exposure time for the accelerated tests is linear
- 2) Available data is limited so that it is not possible to give an allowed range of metallic mass loss in those cases

For the purpose of product qualification, results from standard metal specimen exposure under inservice conditions should be used to estimate the most likely lifetime corrosion load a product may be exposed to during its designed service lifetime. This estimated in-service corrosion load may thereafter be used to estimate the necessary exposure time for a product qualification test. How relevant this equivalent corrosion load approach is for the product to be tested and qualified depends on many factors as the corrosion properties of the materials of the product, the standard metal used for corrosion load estimations, the available in-service corrosion data, and the accelerated test selected for product qualification. But, the better the accelerated test is in simulating in-service corrosion behaviour the more reliable predictions can be made from the results of the test of course.

#### 6 Requirements for test equipment and reproducibility of test results

The requirements for test equipment may vary considerably between different standardized accelerated corrosion tests as are shown in <u>Annex A</u>, where important characteristics of some standardized accelerated corrosion tests are given.

In the choice of test equipment, the requirements set by the test method considered the most suitable and that on reproducibility of test results should be first considered. Of importance in the selection of test equipment is, of course, also the availability of test equipment required for the various tests that can come into question and the cost of test.

NOTE When using simple test methods, it is important to check the test conditions very carefully for obtaining a reasonably high reproducibility. The corrosivity of the test should be checked by standard metal coupon exposure and adjusted so that it falls within the prescribed interval as described in most of the newer standards. However, it is also important to take into account that the correlation between test results and inservice corrosion performance is generally much poorer when a simple highly accelerated test is used than when a more advanced accelerated corrosion test is utilized for the same purpose.

#### 7 Recommended procedures for product qualification

For qualification testing of products or functional units with respect to corrosion resistance the following general procedure is recommended.

- 1) Use the results of an initial risk analysis of potential failure modes of the product or functional unit to be qualified, as described in <u>Annex B</u>, to identify a critical failure mode and associated corrosion process that needs to be evaluated by way of accelerated corrosion testing.
- 2) Select appropriate accelerated corrosion test; see <u>Clauses 3</u>, <u>4</u>, <u>5</u>, <u>6</u> and <u>Annex A</u>.
- 3) Select suitable attribute of the functional unit for use as degradation indicator. From the performance requirement evaluate the lowest tolerable level of this degradation indicator to define failure of the functional unit; see Clause B.2.
- 4) Specify in-service corrosivity and design lifetime corrosion load for the functional unit to be tested. The exposure of metal coupons and determination of the rate of corrosion of those is the preferred method for determining severity classes or classes of atmospheric corrosivity in specific inservice environments; see Clause B.3 and <u>Clause 5</u>.
- 5) Estimate acceptable failure time in the accelerated test from the design lifetime corrosion load for the functional unit. Adopt the principle of equivalent corrosion load as described in <u>Clause 5</u>.
- 6) Perform the test and conclude whether the tested functional unit has a failure time higher than the acceptable. Analyse test specimens also with respect to expected degradation mechanism.
- 7) From the results obtained conclude whether the functional unit shall be considered qualified or not in respect of its corrosion resistance.

How the proposed methodology can be adopted is illustrated in <u>Annex A</u> where some examples on product qualification schemes based on accelerated corrosion testing are given.

#### Annex A

(informative)

## Characteristics of standardized accelerated atmospheric corrosion tests

Warning — In this annex, general characteristics of some standardized accelerated corrosion tests are presented for the purpose of comparing the tests. To conduct the tests, the full text of the standards is needed.

#### A.1 ISO 9227, Corrosion tests in artificial atmospheres — Salt spray tests

The oldest and most wildly used method for laboratory accelerated corrosion testing is the continuous neutral salt spray test as described in e.g. ASTM B117, ISO 9227 and IEC 60068-2-11.

#### A.1.1 Recommended fields of applications

The salt spray methods are all suitable for checking that the comparative quality of a metallic material with or without corrosion protection is maintained. They should not be used for comparative testing to rank different materials relative to each other with respect to corrosion resistance.

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. Depending on the composition of the salt mist there are three different salt spray test methods used for different materials:

- the neutral salt spray test (NSS) applies to metals and their alloys, metallic coatings (anodic and cathodic), conversion coatings, anodic oxide coatings, and organic coatings on metallic materials,
- the acetic acid salt spray test (AASS) is especially useful for testing decorative coatings of copper + nickel + chromium or nickel + chromium,
- the copper-accelerated acetic acid salt spray test (CASS) is useful for testing decorative coatings of copper + nickel + chromium or nickel + chromium; it has also been found suitable for testing anodic coatings on aluminium. However, it should be mentioned that no satisfactory basis for comparison can be derived from this test with regard to the respective quality of nickel + chromium coatings and copper + nickel + chromium coatings, because the reagent used contain copper ions which promote corrosion in the presence of nickel but is without influence on copper.

#### A.1.2 Test conditions

In the continuous salt spray test, the test objects are continuously exposed to a salt containing mist or fog under constant temperature conditions. In the NSS test, the salt solution is an aqueous sodium chloride solution of a concentration of  $50 \, \text{g/L}$  giving a pH of the salt mist solution between 6,5 and 7,2. In the AASS test, glacial acetic acid is added to the  $50 \, \text{g/L}$  sodium chloride salt solution so that the pH of the salt mist solution will become around 3,2. In the CASS test, copper(II) chloride dihydrate (CuCl<sub>2</sub>·2H<sub>2</sub>O) is added to the AASS solution to produce a solution with a CuCl<sub>2</sub> concentration of 0,26 g/L and of pH around 3,2.

### A.1.3 Reproducibility and correlation with in-service exposures with respect to observed damage

There is seldom a direct relation between resistance to the action of continuous 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. Nevertheless, the method described gives a means of checking that the comparative quality of a metallic material, with or without corrosion protection, is maintained.

Salt spray tests are generally suitable as corrosion protection tests for rapid analysis for discontinuities, pores and damages 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.

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 as the corrosion stress during these tests differs significantly from the corrosion stresses encountered in practice.

## A.2 ISO 10062, Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)

Products with e.g. sensitive electrical functions may during storage or operation in indoor locations be affected by atmospheric corrosion dependent on climatic factors as temperature, relative humidity, air velocity and rate of change by temperature and humidity. Additionally, gaseous pollutants may seriously affect the rate of corrosion as well as the occurrence of different corrosion mechanisms. Contaminants on the surface, such as salt, dust, oil and compounds liberated from plastics may also affect the rate and mechanism of corrosion.

Different corrosion promoting gaseous pollutants dominate in different field environments:

- sulphur dioxide and nitrogen oxides in atmospheres influenced by combustion of fossil fuels and in traffic environments,
- hydrogen sulphide in atmospheres in the vicinity of petrochemical and steel industry, decaying organic matter, stagnant waters and animals shelters, and
- hydrogen sulphide and chlorine compounds in the vicinity of pulp and paper industry; if chlorine is used for bleaching

Those gaseous pollutants are known to act as single corrosion promoting factors. But, in atmospheres where more than one gaseous pollutant is present, synergistic effects may be initiated. As a result a considerable increase in the corrosion rate may occur compared to the case when the different gaseous pollutants act as single corrosion promoting factors.

#### A.2.1 Recommended fields of applications

This standard specifies tests which are intended to determine the influence of one or more of those flowing polluting gas(es) under determined conditions of temperature and relative humidity.

The tests apply to metals and their alloys, metallic coatings (anodic and cathodic), metals with conversion coatings, metals with anodic oxide coatings and metals with organic coatings.

#### A.2.2 Test conditions

In the corrosion tests test objects are exposed to a flow of air containing corrosion promoting polluting gases under constant conditions of temperature and relative humidity. The standard specifies six alternative mixtures of polluting gases and two test options regarding choice of temperature and

relative humidity. The polluting gases that are considered in the standard are: sulphur dioxide, hydrogen sulphide, nitrogen dioxide and chlorine.

### A.3 ISO 11130, Corrosion of metals and alloys — Alternate immersion test in salt solution

#### A.3.1 Recommended fields of applications

The alternative immersion test, with or without applied mechanical stress, is particularly suitable for quality control during the manufacture of metals including aluminium alloys and ferrous materials, and for assessment purposes during alloy development.

Depending upon the chemical composition of the test solution, the test may be used to simulate the corrosive effects of marine splash zones, de-icing fluids, and acid salt environments.

The alternate immersion test applies to metals and their alloys, certain metallic coatings (anodic and cathodic with respect to the substrate), certain conversion coatings, certain anodic oxide coatings, and organic coatings on metals.

#### A.3.2 Test conditions

The test consists of immersion of a test specimen stressed (see ISO 7539-1) or unstressed, in a salt solution, followed by withdrawal and a period of drying.

The immersion/drying cycle is repeated at a given frequency for a given period. The extent of attack is then evaluated. For many materials, this provides a more severe corrosion test than simple continuous immersion.

## A.4 ISO 11474, Corrosion of metals and alloys — Corrosion tests in artificial atmosphere — Accelerated outdoor test by intermittent spraying of a salt solution (Scab test)

The accelerated outdoor exposure test described in ISO 11474 involves intermittent spraying of a salt solution to simulate and enhance the environmental stress prevailing at marine test sites. It is based on the so-called VOLVO SCAB method, which has long been used by the automotive industry.

#### A.4.1 Recommended fields of applications

The method is mainly intended for comparative testing and one or more reference materials are therefore always necessary. The results obtained do not permit any far-reaching conclusions on the corrosion resistance of the tested metal in all environments where it may be used. The method described can nevertheless give valuable information on the relative performance of materials in service.

This accelerated outdoor corrosion test applies to organic coatings on metals, metallic coatings (anodic and cathodic), chemical conversion coatings, and metals and their alloys. The method is especially suitable for comparative testing in the optimisation of surface treatment systems.

#### A.4.2 Test conditions

In this method, the corrosion process during outdoor exposure is accelerated by intermittently spraying a solution of sodium chloride (mass fraction 3 %) twice a week, 3-4 days apart, onto the test surface. The test duration is normally six months.

#### A.4.3 Recommended procedures for product qualification

The test is mainly suitable for comparative testing. Its use for qualification of rust protective coating systems is limited due to the fact that the quantitative results of the test vary very much with the climatic outdoor conditions during testing. Tests, for example that are started in northern Europe in the autumn

and finished in the springtime, are generally two to three times more aggressive than tests performed during the summer season due to longer periods of wetting during the winter season compared to the summer season.

## A.5 ISO 14993, Corrosion of metals and alloys — Accelerated testing involving cyclic exposure to salt mist, "dry" and "wet" conditions

This test method involves cyclic exposure of test specimens to a mist of salt solution, to drying conditions, and to periods of high humidity. Its particular advantage over the neutral salt spray test (NSS) lies in its better ability to reproduce the corrosion that occurs in outdoor salt-contaminated environments.

#### A.5.1 Recommended fields of applications

The method is recommended for comparative testing and the results obtained do not permit farreaching 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 employed in the test.

The test applies to: metals and their alloys, metallic coatings (anodic and cathodic), conversion coatings, anodic oxide coatings, and organic coatings on metallic materials.

#### A.5.2 Test conditions

The test objects are successively submitted to a mist of neutral salt solution, to drying conditions at low relative humidity, and to periods of high. No condensation on the test objects should occur during the periods of high humidity. The 8 hours test cycle is repeated a certain amount of times in order to reach the desired corrosivity with respect to corrosion of carbon steel and zinc.

## A.6 ISO 16151, Corrosion of metals and alloys — Accelerated cyclic tests with exposure to acidified salt spray, 'dry' and 'wet' conditions

This standard specifies two test methods A and B, which involve cyclic exposure of test specimens to a mist of acidified salt solution, to drying conditions, and to periods of high humidity.

#### A.6.1 Recommended fields of applications

The particular advantage of the two tests over the neutral salt spray test (NSS) as specified in ISO 9227 lies in their better ability to reproduce the corrosion that occurs in outdoor salt-acid rain environments. They are also useful for evaluating cosmetic corrosion.

The methods are mainly intended for comparative testing and the results obtained do not permit farreaching 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-acid rain environments similar to those employed in the test.

Method A applies to metals and their alloys, metallic coatings (cathodic), anodic oxide coatings, and organic coatings on metallic materials. Method B applies to steel coated with anodic coatings, steel coated with anodic coatings covered with conversion coatings.

#### A.6.2 Test conditions

The test conditions for method A are similar to the test conditions in the standard ISO 14993, except that the neutral salt solution is replaced by an acidified salt solution of pH 3,5 in the first step of the three-steps cycle.

In method B, the test objects are successively submitted to a mist of acidified salt solution of pH 2,5, to drying conditions at low relative humidity, and to periods of high humidity. This 8 hours test cycle is

repeated a certain amount of times in order to reach the desired corrosivity with respect to corrosion of carbon steel and zinc.

## A.7 ISO 16701, 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

This cyclic corrosion test includes two phases. The first phase is 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. The second phase consists of controlled cyclic humidity conditions, where the test object is subjected to an environment which alternates between high humidity and comparative dryness.

#### A.7.1 Recommended fields of applications

The test method has been developed for simulation of corrosion on open surfaces (cosmetic corrosion) and consequently the dry periods are relatively short. As the drying up time in crevices is usually much longer, prolonged drying periods are needed to simulate corrosion phenomena in specimens with crevices in a realistic way. The method is thus preferably recommended for evaluation of corrosion and corrosion protection on open surfaces.

The accelerated laboratory corrosion test applies to metals and their alloys, metallic coatings (anodic and cathodic), chemical conversion coatings, and organic coatings on metals. The method is especially suitable for comparative testing in the optimization of surface treatment systems.

#### A.7.2 Test conditions

The 7 days cycle consist of periods of low relative humidity at 45°C followed by period of high relative humidity at 35°C. Twice a week, a salt solution of pH 4 is sprayed onto the test objects for a total period of 90 min per cycle.

#### A.7.3 Recommended procedures for product qualification

The following recommended procedure for product qualification concerns surface treated exterior automotive parts. In general a six-week test should be sufficient to rank any bare metal (alloy) or a metal protected with 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.

## A.8 ISO 21207, Corrosion tests in artificial atmospheres — Accelerated corrosion tests involving alternate exposure to corrosion promoting gases, neutral salt spray and drying

The standard describes two cyclic corrosion test methods involving a short period of neutral salt spray testing, followed by drying and a longer time period of exposure to an air flow containing corrosion promoting gaseous pollutants at high humidity.

#### A.8.1 Recommended fields of applications

The two test methods defined are intended for use 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.

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.

#### A.8.2 Test conditions

The test objects are first subjected to a short period of neutral salt spray followed by a period of drying. They are then subjected to an air flow containing a mixture of the polluting gases sulphur dioxide and nitrogen dioxide at low levels. A 7 days cycle includes 2 periods of salt spraying, 2 periods of drying and 2 periods of exposure to polluting gases at high humidity.

Test method A simulates a moderately aggressive traffic environment while test method B simulates a more severe industrial or traffic environment. The difference between the tests conditions of methods A and B lies in the concentration of polluting gases.

#### A.8.3 Recommended procedures for product qualification

The test methods are mainly used to simulate the corrosive load 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). They are recommended for the qualification of rust protective coatings as well as to predict the life-time of electronic equipment. These methods are also suitable for comparative testing.

#### **Annex B**

(informative)

## Initial risk analysis of potential failure modes for products or functional units

#### **B.1** Principle

Initial risk analysis is an important step in the planning of a product qualification testing scheme. It entails collecting information on performance and durability of the product or functional unit of the product and its materials. This knowledge base is analysed with respect to the intended application to identify critical functional properties, environmental conditions and associated risks for failure. If it is not possible at this stage to conclude whether the product or functional unit of the product should be qualified or disqualified, qualification testing is performed.

From a practical point of view, but also from an economic viewpoint, an assessment of durability or service life by way of testing has to be limited in its scope and focused on the most critical failure and damage modes.

NOTE 1 If it is not possible to make a reasonable judgement on qualification because reliable tests cannot be found, in-service tests should be started. For products with new materials the general recommendation is to start long-term in-service exposure testing on samples of the material as soon as possible in the development process. In-service testing may require a long time, but the result of it constitutes the definite answer to whether the previous prediction of expected service life was reasonable.

NOTE 2 For functional units, which require high service reliability with respect to functional performance, the statistical variation in service life may be very important. In such cases reliability testing, including the testing of large number of test objects is recommended.

#### B.2 Functional and service life requirements on product and functional

Identify as the first step in the analysis the most important functional attributes of the product or functional unit to be qualified by taking into consideration general end-user or specific product requirements related to functional capability or performance. Formulate in terms of those functional properties thereafter performance requirements for the product or functional unit to be qualified. Defining performance requirement shall be accompanied by an assessment of the economic effects of a functional failure. Based on this, a service life requirement shall be defined or a reliability level that must be maintained for a given number of years be set.

NOTE 3 It may be important to understand the consequences of different failures to define general requirements for performance as failure is defined to occur below that performance level at which satisfactory functioning can not be guaranteed. Thus, if the performance requirements are not fulfilled, the particular product or functional unit are regarded as having failed.

NOTE 4 Performance requirements can be formulated on the basis of optical properties, mechanical strength, aesthetic values or other criteria related to the performance of the product or functional unit and its materials. For failure modes characterised by a gradual deterioration in performance, the consequences of failure may not be very significant shortly after the service life requirement is no longer met. For catastrophic types of failure modes, however, the intended functional capability of the functional unit or some part of it may be completely lost.

#### B.3 Potential failure modes and associated material degradation mechanisms

After failures have been defined in terms of the minimum performance levels set, potential failure and damage modes and important degradation processes associated with those shall be identified.

NOTE 5 In general, there exist many kinds of failure modes for a particular functional unit and the different damage mechanisms, which may lead to the same kind of failure, may sometimes be quite numerous.

Fault tree analysis is a tool, which provides a structure relating failure to various damage modes and underlying chemical or physical changes.

For the purpose of product qualification by testing, select also suitable degradation indicators for the different potential failure modes so that failure and the degradation process causing this failure may be assessed properly.

#### B.4 Critical factors of environmental stress and degradation factors

Assess critical factors of environmental stress or degradation factors associated with the potential failure modes, damage modes and critical degradation processes previously identified. Assess also their expected severity under in-service conditions. Specify thereafter the in-service severity the product or functional unit must resist during its expected service life.

NOTE 6 For the purpose of product qualification, in-service severity representing a worst case may be selected. Alternatively, in-service severity based on the most representative case of in-service environment may be used for product qualification.

NOTE 7 For specifying environmental stress severity with respect to atmospheric corrosivity the classification system described in ISO 9223-ISO 9226 is recommended. Alternatively, the classification system in ISA S71.04 may be used. For assessing atmospheric corrosivity by measurements, the exposure of metal coupons and determination of the rate of corrosion of those, as is described in ISO 9226, is the preferred method.

NOTE 8 In the initial risk analysis, the most important issue is to identify in a qualitative way the most critical in-service conditions and environmental stress factors which may contribute to material degradation and cause failure to occur. It is first at the qualification testing stage specification of the in-service severity has to be made in strictly quantitative terms.

#### **B.5** Risk analysis

Estimate from the information gathered in the analysis described in Clauses B.2-B.4, the risk for failure or unsatisfactory functional performance associated with each of the potential failure and damage modes identified. The estimated risk shall be the point of departure to judge whether a particular failure or damage mode needs to be further evaluated or not. It should also be used as starting point when identifying what kind of testing is needed for qualification of the product or functional unit considered.

NOTE 9 The risk or risk number associated with each potential failure/damage mode identified can be estimated by use of the methodology of FMEA (Failure Modes and Effect Analysis) and FMECA (Failure Modes Effects and Critically Analysis, see e.g. references[4,5] in the Bibliography.

NOTE 10 Literature research should be performed prior to risk assessment to gather relevant data on durability and service life data on similar functional units and materials in the same kind of application as the present. For the same reason durability and service life data on the specific functional unit and materials in other applications and in-service environments as the present should also be gathered.

#### **Bibliography**

- [1] NT ELEC 025 Electrical equipment: Combined damp heat, Steady state, Cold water spraying test
- [2] ISA S71. 04 Environmental conditions for process measurements and control systems: Airborne contaminants (Instrument Society of America, 67 Alexander Drive, P.O. Box 12277, Research Triangle Park, North Carolina 27709, USA, (1985))
- [3] Assessment of corrosion protection classes for inorganic coatings on steel, Nordtest Method NT MAT 003, see www.nordicinnovation.net
- [4] The basics of FMEA, R.E. Mc Dermott, R.J. Mikulak, M.R. Beauregard, R. Mikylak, Productivity Inc, 1996
- [5] ECSS-Q-30-02A, *Space product assurance: Failure modes, effects and criticality analysis (FMECA)*, ESA Publications Division, ISSN 1028-396X
- [6] Environmental Engineering Handbook, with Appendixes A and B, SEES Swedish Environmental Engineering Society, 1997, Handbook can be ordered from www.sees.se
- [7] MIL-STD-810E, *Environmental Test methods and Engineering Guidelines*, July 1989, Department of Defense, Standards, Philadelphia, PA 19111-5094 United States
- [8] IEC 60721, Classification of Environmental Conditions, International Electrotechnical Comission, P.O. Box 131, CH-1211 Geneva 20, Switzerland
- [9] Analysis Techniques for System Reliability Procedure for Failure Mode and Effect Analysis (FMEA), IEC Standard, Publication No 812, 1985
- [10] KÖHL M., CARLSSON B., JORGENSEN G., CZANDERNA A.W.eds. *Performance and Durability Assessment of Optical Materials for Solar Thermal Systems*. Elsevier, 2004
- [11] *Corrosion and climatic effects in electronics*, Risto Hienonen and Reima Lahtinen, VTT publication no. 413, VTT Technical Research Centre of Finland, Espoo 2000