

Peter Eriksson
Bo Carlsson

Accelerated Corrosion Test Involving Alternate Exposure for Hostile Gases, Neutral Salt Spray and Drying

SP Method 2499

Abstract

A SP-method for assessing the corrosion resistance of metallic materials by accelerated testing is described. The method comprise two tests, A and B of different severity, which are both designed to simulate and enhance the environmental influence on a product of exposure to an outdoor climate where exposures to salt-contaminated conditions and of hostile gases from an industrial or a traffic environment occur and may promote corrosion. Test method A simulates a moderately aggressive traffic environment while test method B simulates a more severe industrial or traffic environment.

The corrosion tests are based on test cycles where the test objects are first exposed for salt spraying followed by drying and thereafter by an exposure period in a mixture of hostile gases (NO_2 and SO_2) at a relative humidity of 95%. As a measure of corrosivity, the corrosion rate of metallic copper is utilized. For simulating a certain time of exposure in a traffic or an industrial environment recommended periods of test duration, which correspond to the same extent of copper corrosion in the two tests, are given.

The methods are especially suitable for assessing the corrosion resistance of electronic components.

Key words: atmospheric corrosion, NO_2 , SO_2 , accelerated cyclic test, salt spray, copper coupons

Sveriges Provnings- och
Forskningsinstitut
SPRapport1998:44
ISBN91-7848-751-X
ISSN 0284-5172
Borås 1998

Swedish National Testing and
Research Institute
SPReport 1998:44

Postal adress:
Box 857, SE 501 15 BORÅS
Sweden
Telephone + 46 33 16 50 00
Telefax + 46 33 13 55 02
<http://www.sp.se>

Table of contents

Abstract

Table of contents

Foreword

0. Introduction	5
1. Scope	7
2. Normative references	8
3. Apparatus and Reagents	9
3.1 Exposure of test objects for mixtures of NO ₂ and SO ₂ at a relative humidity of 95% and a temperature of 25 °C	9
3.2 Salt spray testing according to ISO 9227	10
3.3 Drying in standard laboratory climate	10
4. Evaluation of the corrosivity of test	11
4.1 Reference test panels	11
4.2 Arrangement of the reference specimens during testing	11
4.3 Determination of mass loss after testing	11
4.4 Satisfactory performance of test	12
5. Test objects	13
6. Procedure	14
6.1 Test method A	14
6.1.1 Test cycle	14
6.1.2 Salt spray testing followed by drying	14
6.1.3 Exposure for hostile gases followed by drying	14
6.1.4 Test duration	15
6.2 Test method B	15
6.2.1 Test cycle	15
6.2.2 Salt spray testing followed by drying	15
6.2.3 Exposure for hostile gases followed by drying	16
6.2.4 Test duration	16
7. Evaluation of results	17
8. Test report	18
9. Bibliography	19
Appendix A	20

Foreword

The present SP method is the result of two research projects aimed at developing accelerated corrosion tests for service life prediction, especially in the field of electronics.

SP method 2499:A is the result from a larger research project including the following participants: AMP Svenska AB, Autoliv Electronics AB, Combitech Electronics AB, Saab Automobile AB, Scania CV AB, Volvo Truck Corporation, Division of Corrosion Science at the Royal Institute of Technology in Stockholm. For financial support the participating industries and the Swedish National Board for Industrial and Technical Development (NUTEK) are acknowledged.

SP method 2499:B is the result of a development effort, which has been partly financed by Ericsson Radio Systems.

We are grateful for the contributions, both financially and scientifically, given by the project members and we hereby thank all collaborators for their work.

0. 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, therefore, designed to simulate the effects of the most important factors enhancing the corrosion of metallic materials.

The accelerated corrosion test methods, A and B, described in this SP-method are designed to simulate and enhance the environmental influence on a product of exposure to an outdoor climate where exposures to salt-contaminated conditions and of hostile gases from an industrial or a traffic environment occur and 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) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate.
- b) exposure for 120 hrs in a test atmosphere containing a mixture of hostile gases, 1,5 ppm NO₂ and 0,5 ppm SO₂ , at a relative humidity of 95% and at a temperature of 25 °C, followed by drying for 24 hrs in standard laboratory climate.

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

- a) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate,
- b) exposure for 48 hrs in a test atmosphere containing a mixture of hostile gases, 10 ppm NO₂ and 5 ppm SO₂ , at a relative humidity of 95% and at a temperature of 25 °C,
- c) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate
- d) exposure for 72 hrs in a test atmosphere containing a mixture of hostile gases, 10 ppm NO₂ and 5 ppm SO₂ , 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 within which it may be used.

Nevertheless, the methods provide valuable information on corrosion resistance of the product exposed to environments similar to those employed in the test.

1. Scope

This SP method defines two accelerated corrosion test methods to be used in assessing the corrosion resistance of products with metallic materials 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 hostile gases from industrial or traffic air pollution.

The 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 electronic components used in traffic and industrial environments.

2. Normative references

- ISO 8407:1991 *Metals and alloys — Procedures for removal of corrosion products from test specimens*
- ISO 9227:1990 *Corrosion tests in artificial atmospheres — Salt spray tests*
- ISO 10062:1991 *Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)*
- IEC 68-2-60:1997 *Environmental testing - Part 2 - Test Ke: Flowing mixed gas corrosion test*

3. Apparatus and Reagents

3.1 Exposure of test objects for mixtures of NO₂ and SO₂ at a relative humidity of 95% and a temperature of 25 °C

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

The equipment used for testing shall be constructed so that:

- 1) The inner chamber and gas flow system consist of inert materials, e.g. Teflon or glass, to avoid or minimise adsorption of hostile gases on surfaces other than of that of the test panels.
- 2) The air flow and hostile gas injection system are designed to ensure uniform test conditions in the inner chamber or working space of cabinet.

NOTE 1 - 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 hostile gases into a damp air flow to obtain the required concentration. The hostile gases and conditioned air are mixed outside the cabinet. The conditioned air is taken from the outer chamber of the climatic cabinet. The air flow after injection of the hostile gases is then mixed with a flow of recirculated 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 recirculated.

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 air flow through the working space.

- 3) 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, as expressed in terms of corrosion rates of copper metal, the uniformity shall be not less than 5%.

NOTE 2 -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 specified range.

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

NOTE 3 - 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 sulphur dioxide gases may be taken either from pressurised cylinders filled with 1000 ppm high-purity gases in high-purity nitrogen gas or from thermostated permeation tubes.

- 5) For exposure of test panels in the working space, specimens holder shall be used so the test panels do not shield one another or disturb the uniformity of air flow across the chamber
- 6) The temperature, relative humidity, and concentration of sulphur dioxide and nitrogen dioxide in the air flow at the outlet of working space is monitored so that they reflect the true test conditions for test objects.

3.2 Salt spray testing according to 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 $23\text{ °C} \pm 2\text{ °C}$ 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 according to this SP-method, use four reference panels of metallic copper as specified in Swedish Standard SS-5015 (corresponds to ISO Cu-DHP).

The reference specimens should have a dimension of 50 mm x 50 mm x 1 mm.

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

1. mount panel with double-sided tape on flat specimen holder,
2. grind the front surface of panel on paper (220 grit) with water until flat,
3. polish panel for 5 minutes on cloth with 15 μ m diamond suspension and ethanol,
4. remove panel from holder and repeat the steps 1-3 but now with the back side up,
5. stamp identification number on panel,
6. polish panel with 9 μ m diamond suspension and Kleenex manually,
7. clean panel in ethanol and wipe it with Kleenex paper manually,
8. clean panel in ultrasonic bath with ethanol and blow it dry,
9. store the panel in excikator for a minimum time period of 30 minutes,
10. weigh the panel to the nearest 0,1 mg.

4.2 Arrangement of the reference specimens during testing

During testing handle the reference panels in the same way as the test objects. During salt spray testing and exposure testing in the flow of the mixed hostile gases, position the four reference panels 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 remove the corrosion products from the reference panels by repetitive cleaning as described in ISO 8407.

Use for chemical cleaning a 5 weight % solution of HSO_3NH_2 in distilled water. The chemical cleaning procedure is in both cases preferably performed in repetitive steps of immersion of 1 minute at room temperature. After each immersion step, thoroughly clean the reference specimen at ambient temperature by immersion in water, then immersion in acetone or ethanol, followed by drying. To avoid oxidation during chemical cleaning the solutions should be saturated with nitrogen gas prior to and during this process.

Weigh the reference panels to the nearest 0,1 mg and plot the mass versus the actual cleaning cycle as described in ISO 8407.

NOTE 4 - For the efficient dissolution of the corrosion products during the immersion step it is important that the solution is kept stirred. An ultrasonic agitation may preferably be used for increasing the rate of dissolution.

From the plot of mass versus number of cleaning cycles determine the true mass of the specimen after removal of the corrosion products as described in ISO 8407. Subtract this number with the initial mass of the reference panel prior to testing and divide the resulting number with the area of the exposed surface area of the reference panel to assess the metal mass loss per square metre of the reference panel.

4.4 Satisfactory performance of test

The test shall be considered to have been performed satisfactorily if the loss in mass of each reference panel is within the following intervals:

Test method	Test duration	Allowed range of mass loss of reference panel (mg/dm ²)
A	2 weeks	75 ± 6
	4 weeks	145 ± 15
	6 weeks	200 ± 40
B	1 week	100 ± 10
	2 weeks	250 ± 25
	3 weeks	400 ± 40

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 between the interested parties.

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

- a) Specification of product to be tested.
- b) If the test object is subjected to intentional damage, the shape and the location of the damage should be described, as well as how the damage was achieved. The orientation of the damage during testing should also be specified.
- c) Information on cleaning procedure to be used prior to testing
- d) Information on reference material or materials with which the test object is to be compared.
- e) Test duration required
- f) How the test object is to be examined and which properties are to 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) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate, according to what is described in clause 6.1.2
- b) exposure for 120 hrs in a test atmosphere containing a mixture of hostile gases, 1,5 ppm NO₂ and 0,5 ppm SO₂, at a relative humidity of 95% and at a temperature of 25 °C, followed by drying for 24 hrs in standard laboratory climate, according to what is described in clause 6.1.3.

6.1.2 Salt spray testing followed by drying (step a)

6.1.2.1 Place the test objects and the reference panels in the salt spray cabinet and expose them for a mist of a 5 % sodium chloride salt solution at 35 °C according to ISO 9227. After 1 hour 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 hours of testing, the test objects shall be removed from the cabinet and brought to a constant climate room without first being subjected for rinsing.

6.1.2.2 Store the test objects and reference panels in the constant climate room at 23 °C and 50% RH, for 22 hours before initiating step b) described in clause 6.1.3

6.1.3 Exposure for hostile gases followed by drying (step b)

6.1.3.1 Prepare the climatic cabinet for test by firstly adjusting temperature to 25 °C, air humidity to 95% RH and air flow rate at a selected value between 0,5 mm/s and 5 mm/s. After stable conditions have been reached, adjust the hostile gas flows so that the sulphur dioxide concentration in the inlet air flow to the working space will be at a level of 0,5 ppm and the nitrogen dioxide concentration in the inlet air flow to the working space will be at a level of 1,5 ppm .

6.1.3.2 When stabilised conditions have been reached also after this step, open the door to cabinet and place the test objects and reference panels quickly in the working space. After a testing time period not more than 6 hrs, the concentration of sulphur dioxide and nitrogen dioxide in the outlet air flow from the working space shall not be less than 80% of that in the inlet flow of air. If the concentration of the hostile gases in the outlet air flow is lower, this probably means the total area of test objects in the cabinet is too large. During the test, check the exposure conditions regularly and, if necessary, make adjustments to the specified levels.

6.1.3.3 After 120 hrs of test remove the test objects from the working space of cabinet and without subjecting them for any cleaning procedure perform as described in clause 6.1.2. If the test is completed place the reference panels in a constant climate room prior to weighing.

6.1.3.4 Store the test objects and reference panels in the constant climate room at 23 °C and 50% RH, for 24 hours before initiating step a) described in clause 6.1.2.

6.1.4 Test duration

One test cycle corresponds to one weeks 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 by the interested parties.

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) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate, according to what is described in clause 6.2.2.
- b) exposure for 48 hrs in a test atmosphere containing a mixture of hostile gases, 10 ppm NO₂ and 5 ppm SO₂ , at a relative humidity of 95% and at a temperature of 25 °C, according to what is described in 6.2.3
- c) traditional salt spray testing (ISO 9227) for 2 hrs in a mist of a 5 % sodium chloride salt solution at 35 °C, followed by drying for 22 hrs in standard laboratory climate, according to what is described in clause 6.2.2
- d) exposure for 72 hrs in a test atmosphere containing a mixture of hostile gases, 10 ppm NO₂ and 5 ppm SO₂ , at a relative humidity of 95% and at a temperature of 25 °C, according to what is described in 6.2.3

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

6.2.2.1 Place the test objects and the reference panels in the salt spray cabinet and expose them for a mist of a 5 % sodium chloride salt solution at 35 °C according to ISO 9227. After 1 hour 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 hours of testing, the test objects shall be removed from the cabinet and brought to a constant climate room without first being subjected for rinsing.

6.2.2.2 Store the test objects and reference panels in the constant climate room at 23 °C and 50% RH, for 22 hours before initiating step b) or d) described in clause 6.2.3

6.2.3 Exposure for hostile gases (step b or step d)

6.2.3.1 Prepare the climatic cabinet for test by firstly adjusting temperature to 25 °C, air humidity to 95% RH and air flow rate at a selected value between 0,5 mm/s and 5 mm/s. After stable conditions have been reached, adjust the hostile gas flows so that the sulphur dioxide concentration in the inlet air flow to the working space will be at a level of 5 ppm and the nitrogen dioxide concentration in the inlet air flow to the working space will be at a level of 10 ppm.

6.2.3.2 When stabilised conditions have been reached also after this step, open the door to cabinet and place the test objects and reference panels quickly in the working space. After a testing time period not more than 6 hrs, the concentration of sulphur dioxide and nitrogen dioxide in the outlet air flow from the working space shall not be less than 80% of that in the inlet flow of air. If the concentration of the hostile gases in the outlet air flow is lower, this probably means the total area of test objects in the cabinet is too large. During the test, check the exposure conditions regularly and, if necessary, make adjustments to the specified levels.

6.2.3.3 After 48 hrs of test if step b) is performed or after 72 hrs of test if step d) is performed remove the test objects from the working space of cabinet and without subjecting them for any cleaning procedure. Proceed thereafter to step c) if the test duration was 48 hrs or to step a) if the test duration was 72 hrs. If the test is completed place the reference panels in a 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 by the interested parties.

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

NOTE 5 - It is recommended 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, for example:

- a) appearance after the test;
- b) number and distribution of corrosion defects, i.e. pits, cracks, blisters, etc.; these may be assessed by methods described in ISO 1462 or ISO 4540;
- c) the time elapsing before the appearance of the first signs of corrosion
- g) change in mechanical or electrical properties;

NOTE 6- It is good engineering practice to define the appropriate criteria in the specification for a product to be tested.

8. Test report

The test report shall provide the following information:

- a) reference to this SP-method;
- 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 5.2 of this SP-method, 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;
- g) the number of cycles or the duration of the test;
- h) the frequency and intervals of inspection during test;
- i) the corrosivity of test determined as described in clause 4;
- j) any deviations from the prescribed testing method;
- k) test results after final evaluation of test objects;

9. Bibliography

- [1] Test methods for environmental resistance of electric components in a traffic environment - Case study on corrosion resistance of electronics and contacts for automobiles Peter Eriksson et al; SP-Report to be published
- [2] "UN/ECE international co-operative programme on effects on materials, including historic and cultural monuments", Report No.11: Corrosion attack on copper and cast bronze. Evaluation after 4 years of exposure, Bavarian State Conservation office, Munich, Germany (1993).

Appendix A

Table A1 shows recommended periods of test duration for simulating the corrosive load after certain years of exposure in a traffic environment (Test method A) and in a more severe traffic environment or industrial environment (Test method B).

The recommended periods of test duration for test method A origin from a field study on copper corrosion on vehicles [1]. 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 [2].

Table A1 — Recommended periods of test duration
(the values are based on the corrosion rate of copper).

Test method			
	Type of environment	Years in specified environment	Corresponding test duration
A	Traffic	5	3 weeks
		10	6 weeks
B	Industrial	3	1 week
		8	2 weeks
		14	3 weeks