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Neutral salt spray test — Results of an international interlaboratory test and conclusions for practical application

Essai au brouillard salin neutre — Résultats d'un essai interlaboratoire international et conclusions pour une application pratique

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Foreword

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This document was prepared by Technical Committee ISO/TC 2, *Fasteners*, Subcommittee SC 14, *Surface coatings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

0.1 General

The neutral salt spray test (NSS test) has been used for at least one century for the corrosion evaluation of various protective layers, including coatings for fasteners. This test is well-known and used worldwide by all involved parties (chemical suppliers, job coaters, manufacturers of components including fasteners, purchasers ...), due to its relatively short testing time compatible with industrial production.

The NSS test is generally considered a suitable method for determining the effectiveness of corrosion protection. The ability to expose samples and components quickly makes it possible to identify weaknesses, pores and damages in organic and inorganic coatings. It is to be noted here that by the use of a salt spray with a defined composition (5 % sodium chloride, NaCl) and by the material-related formation of protection layers there is no direct correlation to other media capable of inducing corrosion. Instead, the results obtained using this method serve to compare different surface conditions under specified and constant conditions. As such, the salt spray test is an effective tool for the quality control of products exposed to corrosive operating conditions.

In terms of the reproducibility of the test results, it is beneficial if the coatings and layers are sufficiently similar. In addition, there are numerous manufacturers that offer different types of salt spray cabinets. The various construction concepts available on the market, as well as factors such as the loading of the cabinet or the use of samples with different geometries, influence the salt spray formation inside the cabinet. This makes it difficult to conduct a comparative assessment of the corrosion test results obtained using different test equipment. For this reason, a project group was established within the Working Group Surface Protection Coating Systems of Deutscher Schraubenverband e.V. with the objective of compiling salt spray test results from as many laboratories as possible and then preparing a comparative assessment.

The samples consisted of bolts that were electrolytically coated using two different zinc-based variants. A total of 39 participants, consisting of bolt manufacturers, job coaters, users and institutes with 75 cabinets from 11 countries were involved in the tests, see Annex A. The large number of participants from all sectors throughout the value chain of a coating process enables a reliable statistical analysis of the test results.

0.2 Executive summary and conclusion

One objective of the interlaboratory test was to conduct a neutral salt spray test in accordance with ISO 9227 on two coating variants of bolts M6×50. Time of occurrence of gray veil, white rust and red rust was documented. The corrosivity of the salt spray was determined by means of assessment of the mass loss of an uncoated steel panel as specified in ISO 9227 as well as the determination of the time until appearance of red rust on hot-dip galvanized steel panels in accordance with ISO 4042 and ISO 10683.

Another objective was to compare the two methods used to determine the corrosivity of the salt spray in order to establish the suitability of these methods by comparing the corrosion assessment results obtained for the bolts.

In addition, the normative operating parameters (temperature in the test cabinet, collection rate, pH and density or NaCl concentration of the solution collected) were documented to ascertain whether there is any correlation with the results of the corrosion assessment performed on the bolts.

The main objective of the interlaboratory test was the determination of the reproducibility of the salt spray test.

A statement has been included in the introduction of the current version of ISO 9227:

“When interpreting test results (e.g. minimum time to damage or corrosion) for product quality control or acceptance specifications, it is important to note that salt spray testing may have low reproducibility, especially for manufacturing parts that are tested in different laboratories.”

These findings are supported by the interlaboratory study. The results are summarized as follows:

— The mass losses of the standardized test panels as per ISO 9227 and the corrosion behaviour of standardized, hot-dip galvanized test panels as per ISO 4042 do not correlate with the corrosion behaviour observed on the zinc-electroplated and zinc-nickel electroplated bolts M6x50 with transparent or black passivation that were tested in parallel.

— There seems to be no clear connection between the criterion of “compliance with the normative test parameters as per ISO 9227”, which is used to classify the corrosion cabinets as “compliant” or “non-compliant”, and the assessed corrosion behaviour of the test panels or the corrosion behaviour of the bolts tested. Accordingly, compliant operation in line with normative test parameters does not lead to a reduced scatter of the times to failure recorded for the selected coated bolts examined as part of this interlaboratory test.

Additional information about the context to understand these results can be found in Clause 8.

Alternatives to salt spray testing, such as cyclic testing procedures, are established in the market but are mostly customer specific: they need different testing equipment and/or dedicated settings for each type of test (environmental cabinets also need specific skills and experts). Such cyclic tests are useful, however, according to the experience of the committee members, do neither solve the question of correlation between the cabinet parameter settings and the results on tested samples nor of observed scattering of the results.

It is the opinion of the experts that salt spray testing in accordance with ISO 9227 and ISO 4042 should still be applied for:

— production process monitoring and verification for the coating process (but not for process control, especially if based on a statistic approach)

— comparison with different parts using the same coating and the same coating process.

Salt spray testing is however not exhaustive and advantageously accompanied by other tests specified in relevant standards.

Neutral salt spray test — Results of an international interlaboratory test and conclusions for practical application

1 Scope

The objective of this document is to conduct a neutral salt spray test in accordance with ISO 9227. The test is a proven method for assessing the corrosion protection of coatings of components such as bolts. For this reason, two coating variants were chosen for conducting the tests on hexagon bolts with a size of M6 × 50. The bolts were examined at specified points in time and the time of occurrence of grey veil, white rust and red rust was documented. The corrosivity of the salt spray was determined by means of two methods and also documented in an evaluation form. These two methods are the assessment of the mass loss of an uncoated steel panel as specified in ISO 9227 as well as the determination of the time until appearance of red rust on hot-dip galvanized steel panels in accordance with ISO 4042 and ISO 10683.

The processing steps are specified in detail in a manual that was provided to the test participants. If followed precisely, these instructions allow for a comparative analysis of the results from the individual labs and make it possible to determine the reproducibility of the salt spray test. Another objective is to compare the two methods used to determine the corrosivity of the salt spray in order to establish the suitability of these methods by comparing the corrosion assessment results obtained for the bolts. In addition, the normative operating parameters (temperature in the test cabinet, collection rate, pH and density or NaCl concentration of the solution collected) were documented for every inspection date in order to ascertain whether there is any correlation with the results of the corrosion assessment performed on the bolts.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Symbols and abbreviated terms

4.1 Symbols

$F(t)$	Weibull distribution function
b	Shape parameter or Weibull slope
e	Exponential function
k_1	Factor for z_U score
k_2	Factor for z_U score

m	Hampel estimator
pH value	Potentia hydrogenii
Ra value	Arithmetic mean roughness
s_R	Reproducibility standard deviation
s_r	Repeatability standard deviation
T	Characteristic life time
t	Time to failure
t_0	Displacement parameter (failure-free time)
t_{10}	Time to 10 % probability of failure
z_U score	Score for the assessment of a laboratory mean value

4.2 Abbreviations

ASTM	American Society for Testing and Materials
DIN	German Institute for Standardization (Deutsches Institut für Normung)
DSV	Deutscher Schraubenverband e. V. (German Fastener Association)
D0–D4	Scale for the degree of coverage
EN	European Standard
G0–G4	Gray veil scale
ISO	International Organization for Standardization
M	Metric thread
n	Lab/cabinet No.
NaCl	Sodium chloride
XRF	X-ray fluorescence spectroscopy
W0–W4	White rust scale

5 Test samples

The following test and reference samples were used, Figure 1 to Figure 4:

Corrosion test panels in accordance with ISO 4042 or ISO 10683:2018, Figure 1:

Material:	CR24 as per ISO 6932
Dimensions:	190 mm × 90 mm
Surface condition:	Hot-dip galvanized, (11 ± 1) µm

Corrosion test panels in accordance with ISO 9227:2022, Figure 2:

Material: CR4 as per ISO 3574

Dimensions: 150 mm × 70 mm

Surface condition: Uncoated, dull, degreased, roughness *Ra* = (0,8 ± 0,3) µm

Test bolts silver coloured, Figure 3:

Material: Quenched and tempered steel

Dimensions: M6 × 50

Surface condition: Zinc electroplated, thick-film passivated, sealed

Coating thickness: 9,3 µm (min. 9,0 µm, max. 9,6 µm)

Test bolts black coloured, Figure 4:

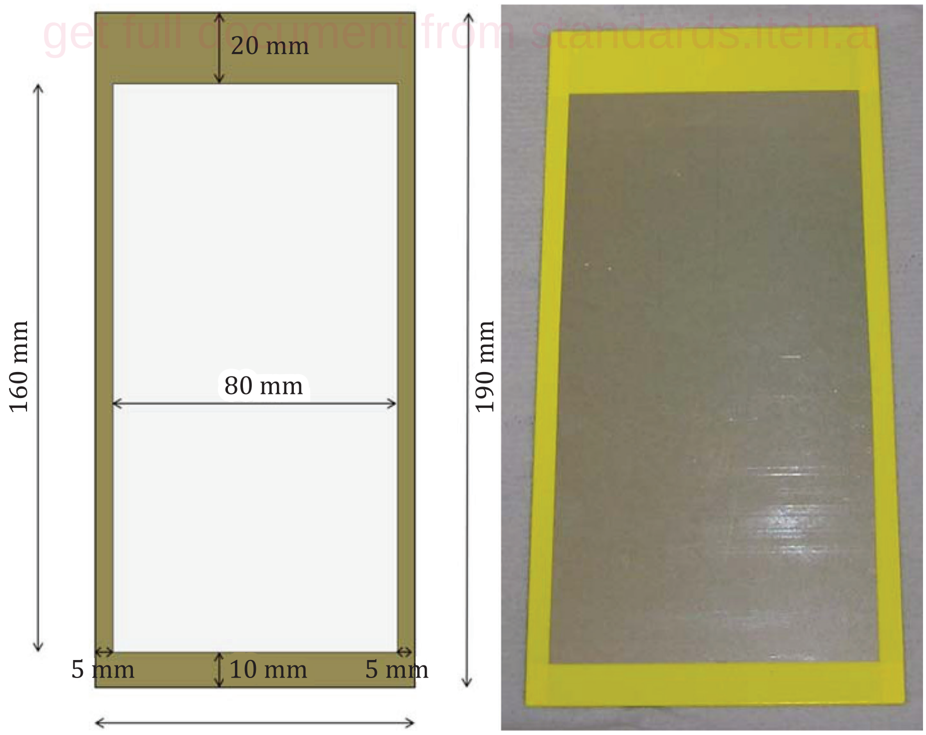
Material: Quenched and tempered steel

Dimensions: M6 × 50

Surface condition: Zinc-nickel electroplated, black passivated, sealed

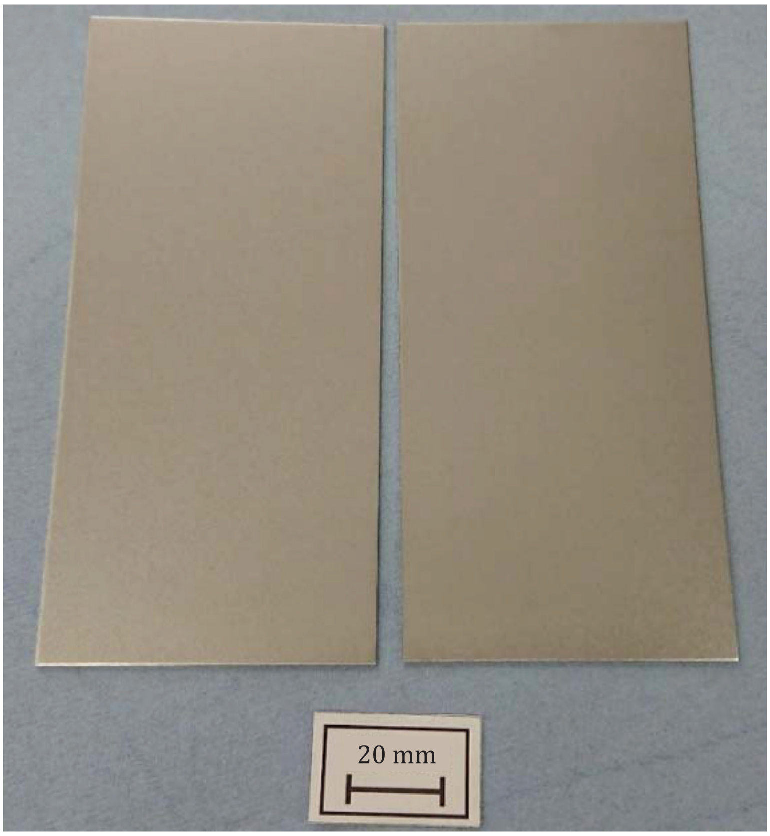
Coating thickness: 9,0 µm (min. 8,2 µm, max. 9,6 µm)

The coating thickness of the test bolts was determined for 10 samples each at the point of force application on the bolt head using X-ray fluorescence spectroscopy (XRF) in accordance with ISO 3497. The thickness of the sealers was not determined. All test samples were shipped in shrink-wrap packaging in order to prevent any prior corrosive damage and reduce the risk of impact damage to the films. The cleaning solution was also shipped in powder form. The participants cleaned and masked the hot-dip galvanized panels themselves.



[SOURCE: Atotech Deutschland GmbH & Co. KG]

Figure 1 — Hot-dip galvanized corrosion test panel in accordance with ISO 4042



[SOURCE: TU Darmstadt]

Figure 2 — Corrosion test panel in accordance with ISO 9227

[SOURCE: TU Darmstadt]

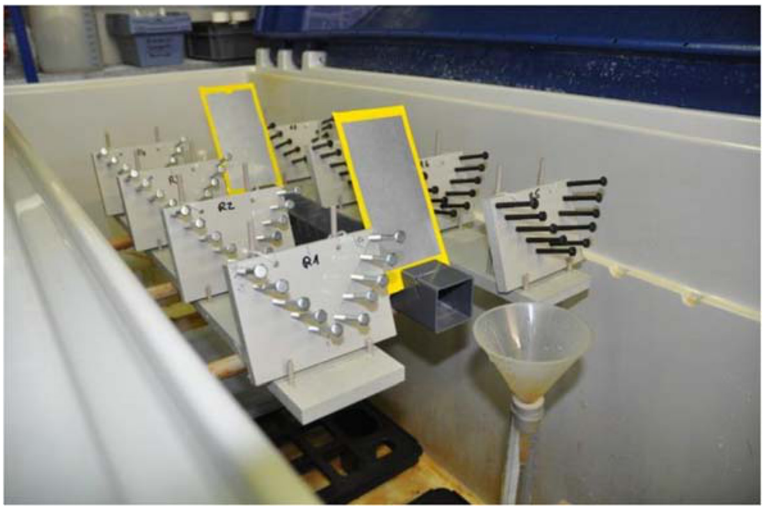
Figure 3 — Silver coloured test bolts: zinc electroplated, thick-film passivated and sealed

[SOURCE: TU Darmstadt]

Figure 4 — Black coloured test bolts: zinc-nickel electroplated, black passivated and sealed

6 Test procedure

Prior to conducting the tests, the degree of coverage of the head and the non-threaded shank of the black bolts was determined and documented in accordance with DIN 34804 on assessing the appearance of black surface coating systems. From the 30 bolts supplied, 20 bolts were selected based on subjective evaluation, with the coating surface as intact as possible. The normative operating parameters of the cabinet, the test panels and the test bolts were inspected and assessed during the test and the results documented. If possible and unless specified otherwise (see 7.2), the operating parameters and the bolts were inspected every 24 h. This inspection could be skipped on weekends and public holidays. The following chapters provide a detailed overview of the inspection intervals. The results were entered in the report table provided by the test organizers. The participants made sure that the cabinet was not left open for more than 1 h during the inspections. The corrosion test panels and the test bolts were inserted into the cabinet at an angle of 20° to the vertical axis. The use of suitable holding devices was not mandatory, but recommended. A possible positioning of the collectors and the test panels and test bolts can be seen in Figure 5.



[SOURCE: TU Darmstadt]

Figure 5 — Example of a 1 000 l cabinet equipped with plastic panels with M6 threaded holes and bolts at the beginning of the test

The test panels were positioned in the same area of the salt spray cabinet as the test bolts. In addition, the other specifications of ISO 9227 applied in order to ensure a freely circulating salt spray in the cabinet. This also included observing the specified minimum distance to the wall. The samples were not positioned directly in line of the spray jet and did not shield one another so that the salt solution film on the surface could not drip from one sample onto another. Furthermore, only the samples to be tested were positioned freely in the salt spray cabinet, if possible. To accommodate the participants' daily operating processes, however, it was also acceptable to insert other samples that were not part of the interlaboratory test into the test cabinet.

6.1 Normative operating parameters

The documentation of the normative operating parameters included the temperature in the test cabinet, the amount of salt solution accumulated in the two collectors as well as the pH value, density or NaCl concentration of the solution. In addition, the parameters of the test solution (conductivity of the water used, pH value, density or NaCl concentration) were recorded and entered into the report table prior to using the solution in the cabinet. This also applied to all test solutions replenished into the salt spray cabinet over the entire course of the test.

6.2 Corrosion test panels

6.2.1 Evaluation in accordance with ISO 4042

At the beginning of the interlaboratory test, the hot-dip galvanized test panels were subjected to salt spray testing for 120 h in accordance with ISO 4042. Prior to testing, the panels were cleaned using the provided cleaning agent in line with the test instructions and then masked, Figure 1. Finally, the panels were positioned in the cabinet at an angle of 20°, see Figure 5, and the test started within 24 h.

The first assessment of the panels took place after 72 h. The cabinet remained closed during the first 72 h, i.e., the operating parameters (see 7.1) were not recorded after 24 h and 48 h and the bolts were not checked. At the inspection intervals (after 72 h, 96 h and 120 h), the panels were assessed in a wet, unrinsed condition. To this end, the delivered control mask was placed on the panel, Figure 6, and all boxes with visible red rust were counted and documented in relation to the total number of boxes. The time in the assessment at which at least 7 boxes (5 %) show visible signs of red rust was also documented in the report table. If fewer than 7 boxes were counted after 120 h, the value ">120 h" was entered in the table.