INTERNATIONAL STANDARD

ISO 11130

Third edition
2017-12

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

Corrosion des métaux et alliages — Essai en immersions alternées en solution saline

Reference number
ISO 11130:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 11130:2010), which has been technically revised with the following changes:

- harmonization with ISO 9227;
- revision of the temperature and relative humidity of drying conditions.

Introduction

Corrosion of metals is influenced by factors which can vary significantly with environmental conditions. Therefore, corrosion resistance determined for metals during alternate immersion testing as described in this document can vary greatly with the test solution selected, the temperature during immersion and the temperature and humidity during the drying periods of the test.

Consequently, the result of an alternate immersion corrosion test is not taken as an indication of the corrosion resistance of the metal tested in all the different service environments where the metal can be used.

Nevertheless, results obtained by the method described in this document can indicate the relative corrosion resistance of different metals under in-service conditions, in particular when the service environment is similar to the test solution selected. The method can also be used to test metals under an applied tensile stress.

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

1 Scope

This document specifies a method for assessing the corrosion resistance of metals by an alternate immersion test in salt solution, with or without applied stress.

The test is particularly suitable for quality control during the manufacture of metals including aluminium alloys and ferrous materials, and also for assessment purposes during alloy development.

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

The term “metal” as used in this document includes metallic materials with or without corrosion protection.

This document is applicable to

- metals and their alloys,
- certain metallic coatings (anodic and cathodic with respect to the substrate),
- certain conversion coatings,
- certain anodic oxide coating, and
- organic coatings on metals.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

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

The immersion and 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.

5 Test solution

5.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or deionized water or water of equivalent purity.

The test solution shall be prepared in accordance with the prescribed specification. Otherwise, the solution used should be the one most appropriate to the intended service conditions. Details of a neutral salt solution that is suitable for simulating the corrosive effect of a marine environment are given in 5.2.

Details of three other test solutions suitable for simulating salt-based de-icing liquid, acid salt conditions and ocean water are given in Annex A.

5.2 Preparation

The neutral salt solution is prepared by dissolving a sufficient mass of sodium chloride in water to give a concentration of 35 g/l ± 1 g/l. The water used shall have a conductivity not higher than 2 mS/m (equal to 20 µS/cm) at 25 °C ± 2 °C.

The sodium chloride shall contain a mass fraction of the heavy metals of copper (Cu), nickel (Ni) and lead (Pb) in total less than 0,005 %. It shall not contain a mass fraction of sodium iodide more than 0,1 % and a mass fraction of total impurities more than 0,5 %, calculated for dry salt. Sodium chloride with anti-caking agents should not be used, because such agents may act as corrosion inhibitors or accelerators.

NOTE A useful sodium chloride salt grade is a grade named Ph. Eur/USP or JIS, ACS.

Prior to use, check the pH of the salt solution using electrometric measurement. Measurements of pH shall be done using electrodes suitable for measuring pH in weakly buffered sodium chloride solutions in deionized water. Make any necessary corrections by adding hydrochloric acid, sodium hydroxide or sodium bicarbonate solution of analytical grade.

The volume of the test solution shall be defined by the specification. If no specification is available, it is recommended that the volume should be not less than 3 l per square decimetre of test specimen area.

6 Apparatus

6.1 General

The apparatus shall include the following components.

- A suitable system designed for the automatic, continuous performance of complete cycles of alternate immersion and withdrawal of the test specimens. This system shall provide uninterrupted operation throughout the duration of the test (see 8.1). Each test specimen shall be connected to the system using suitable insulating material.
- One or more specimen cabinet for the test solution. Only one kind of metal, alloy or coating shall be immersed in each specimen cabinet. Replicate specimens can share the same specimen cabinet.

The system shall be designed such that the time taken for full immersion or withdrawal of each specimen is not more than 2 min.

NOTE Suitable apparatus for conducting alternate immersion tests in salt solution are illustrated schematically in Annex B.

6.2 Materials of construction

6.2.1 Materials of construction that come into contact with the test solution shall be such that they are not affected by the corrodent to the extent that they can cause contamination of the solution and change its corrosivity.

6.2.2 Use of inert materials is recommended where feasible.

6.2.3 Metallic construction materials shall be selected from metals or alloys that are corrosion-resistant to the test environment or shall be protected with a suitable corrosion-resistant coating that also satisfies the conditions given in 6.2.1.

6.3 Test specimen holders

6.3.1 Test specimen holders shall be designed to insulate electrically the test specimens from each other and from any other bare metal. When this is not possible, as in the case of certain stressing bolts or jigs, the bare metal in contact with the specimen shall be isolated from the corrodent by suitable insulating materials. If a protective coating is used, it shall be of a type that will not leach inhibiting or accelerating ions or protective oils over the non-coated portions of the specimen. In particular, coatings containing chromates shall be avoided.

6.3.2 The shape and form of test specimen supports and holders shall be such that

- they avoid, as much as possible, any interference of free contact of the test specimen with the salt solution,
- they do not obstruct air flow over the test specimen, thereby retarding the drying rate, and
- they do not retain a pool of solution in contact with the test specimen after withdrawal from the solution.

6.4 Air circulation

6.4.1 Air circulation is recognized as an important factor because it affects both the rate at which test specimens dry and the loss of water by evaporation. Optimum conditions for air circulation have not been established.

6.4.2 It is important to provide uniform drying conditions for test specimens. A circulation of air capable of drying specimens within about 40 min is recommended.

Drying by forced air blasts on the test specimens is not recommended because of the difficulty in maintaining uniform drying of large groups of test specimens. Moreover, stagnant air conditions shall be avoided.

7 Test specimens

The test shall be performed with manufactured products or parts or with any other suitable test specimens.

The shape and dimensions of test specimen should be agreed between the interested parties. If no agreement is made, rectangular test specimen of 90 mm × 120 mm × 1 mm is recommended.

A minimum of three test specimens should be used.

If the size of the test specimens is incompatible with the specimen cabinet, the specimens shall be sectioned. The cut edge should be protected by a suitable coating applied to a distance of 5 mm around the cut. Under these circumstances, for comparison purposes, six specimens shall be tested, three with and three without protection.

All greases should be carefully removed from the specimens by means of an appropriate method, for example ultrasonic or manual cleaning using a soft, clean brush in a vessel filled with suitable organic solvent (e.g. hydrocarbon with a boiling point of 60 °C to 120 °C). After cleaning, the specimens shall be rinsed using clean solvent then dried.

If a plated or coated test specimen has to be cut, the cut edges shall be protected.

8 Procedure

8.1 Test conditions

Generally, the test conditions are prescribed in the agreed specifications. If this is not the case, the exposures should involve a 10 min immersion followed by withdrawal and a 50 min drying period.

The cycle shall be repeated continuously throughout the duration of the test unless prior failure occurs.

The solution temperature should be 25 °C ± 2 °C, unless otherwise specified. Other solution temperature, i.e. 40 °C, 50 °C, 60 °C, may be used by agreement between the interested parties. It shall be stated in the test report.

The temperature of drying condition should be 70 °C ± 2 °C and the relative humidity should be not more than 50 %, unless otherwise specified. Other temperature and relative humidity may be used under the drying condition by agreement between the interested parties. It shall be stated in the test report.

Only one kind of metal, alloy or coating shall be immersed in the same specimen cabinet during a particular test.

Unless otherwise specified, the test duration shall be established on the basis of the susceptibility to corrosion of the metal in the test solution and the purpose of the test.

NOTE Test duration in the range 20 days to 90 days is usually adequate for aluminium alloys and ferrous metals.

8.2 Immersion

The test specimen shall be immersed in the test solution completely under the depth of at least 10 mm from the solution surface.

The level of test solution in the test cabinet shall be maintained by the addition of deionized water to replenish evaporative losses as required.

The solution shall be changed every 168 h, the pH shall be reported at the beginning and the end.

9 Cleaning of test specimens

On completion of testing, test specimens are removed from the specimen cabinet and cleaned as thoroughly as possible in order to prevent further corrosion. A suitable method is to rinse the test specimens gently in clean running tap water, at a temperature not exceeding 40 °C, and then to dry them immediately in a stream of air, at an overpressure not exceeding 200 kPa and at a distance of approximately 300 mm.

10 Assessment of results

Many criteria may be used to evaluate results according to the particular requirements of the test, such as the following:

a) the appearance after the test;

b) the appearance following the removal of superficial corrosion products;

c) the number and distribution of corrosion effects, i.e. pits, cracks, blisters, etc.; these may be assessed by methods such as those specified in ISO 10289 or the relevant part of the ISO 4628 series;

d) metallographic examination to detect cracks in stressed samples using low-power microscopy at the standard magnification or, typically, ×20;

e) the time elapsed before the appearance of the first sign of corrosion;

f) the change in mass (in accordance with ISO 8407);

g) changes revealed by microscopic examination;

h) changes in mechanical properties;

i) the average and maximum depths of attack.

NOTE It is good engineering practice to specify the assessment criteria in the specification for a coating or a product to be tested.

11 Test report

11.1 The test report shall indicate the outcome of the test according to the evaluation criteria prescribed for the test (see Clause 10). The results obtained for each test specimen shall be reported and, when appropriate, the averages for a group of replicate test specimens. The report may, if required, be accompanied by photographic records of the specimens.

11.2 The test report shall provide information about the test procedure. This may vary according to the purpose of the test and to the test specification but, in general, the details likely to be included are as follows:

a) a reference to this document, i.e. ISO 11130:2017;

b) the specification (or composition) of the material, and its heat treatment;

c) the characteristics of any coating, with an indication of the surface area;

d) details of any applied stress and mode of stressing;

e) the specification of the test cycle, especially the information about the time period of immersion and that of drying;

f) the composition of test solution and purity of water and reagents;

g) the temperature of the solution, the temperature and relative humidity of circulating air used for drying during the test;

h) the pH of the solution at the beginning and the end every 168 h;

i) the shape and dimensions of the test specimen and the nature and area of the surface tested;

j) the preparation of the test specimen, including any cleaning treatment applied, and any protection given to the edges or other special areas;

k) the number of test specimens of each material or product subjected to the test;

l) the method used to clean test specimens after the test with, where appropriate, an indication of the loss in mass resulting from the cleaning operation;

m) the angle of inclination of the tested surfaces;

n) the frequency and number of any test specimen location permutations;

o) the test duration and the results of any intermediate inspections;

p) the properties of any test specimens placed in the specimen cabinet to validate the operating conditions;

q) any deviations from the procedure;

r) any unusual features observed;

s) the inspection intervals;

t) the date of the test.

Annex A
(informative)

Suggested test solutions

WARNING — Safety rules for personnel: handling of the reagents used for preparation of test solutions shall be left to skilled personnel or conducted under their control. The equipment shall be used and maintained by skilled personnel, not only so that the procedures can be performed correctly, but also because of the hazards to health and safety that are involved.

A.1 Test solution for simulating the corrosive effects of de-icing solution

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or deionized water or water of equivalent purity.

A.1.1 Reagents for preparation of the solution

Sodium sulfate, anhydrous (Na_2SO_4)	0,500 g ± 2 mg
Sodium sulfite, anhydrous (Na_2SO_3)	0,250 g ± 2 mg
Sodium thiosulfate, anhydrous ($Na_2S_2O_3$)	0,100 g ± 2 mg
Sodium chloride (NaCl)	52,5 g ± 1 g
Calcium chloride dihydrate ($CaCl_2{\cdot}2H_2O$)	52,5 g ± 1 g
Water	

The pH of the water should not be less than 5,5. If it is lower, boil and cool to room temperature (25 °C) before using.

The above reagents should be stored in a dessicator over calcium sulfate.

A.1.2 Preparation of the solution

a) Weigh out all reagents to the designated accuracy.

b) Add 525 ml of water to a clean 3 l beaker and slowly add the first four reagents in the order listed above, (i.e. sodium sulfate, sodium sulfite, sodium thiosulfate and sodium chloride). Each reagent shall be dissolved and thoroughly mixed before the next reagent is added. Label this as solution A.

c) Add 525 ml of water to a second clean 3 l beaker and slowly add the calcium chloride. Dissolve and thoroughly mix the solution until it is clear and colourless. Label this as solution B.

d) Slowly add solution B to solution A, stirring continuously. If this operation is conducted too rapidly, a precipitation of calcium sulfite may occur. If a precipitate forms, gravity-filter through a cotton ashless filter[1] with a pore size 2,5 µm (slow filter paper) flowrate 1 870 s/100 ml and discard.

e) Adjust the pH of the solution to 9,3 ± 0,5 immediately after preparation using diluted NaOH or diluted HCl. Determine the pH of the solution by electrometric measurement at 25 °C ± 2 °C.

---

[1] No. 42 Whatman filter paper is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

f) Shelf-life of the test solution:

  1) store the solution in coloured (or brown) glass-stoppered bottles;

  2) the solution can be used within 8 days following preparation, with proper adjustment of the pH being made just prior to use in the test programme.

A.2 Test solution for simulating the corrosive effects of acidic salt solution

A.2.1 Preparation of the sodium chloride solution

Dissolve a sufficient mass of sodium chloride in distilled or deionized water with a conductivity not higher than 2 mS/m (equal to 20 µS/cm) at 25 °C ± 2 °C to produce a concentration of 50 g/l ± 5 g/l. The specific gravity range for a 50 g/l ± 5 g/l solution is 1,029 g/cm^3 to 1,036 g/cm^3 at 25 °C.

NOTE A useful sodium chloride salt grade is a grade named Ph. Eur/USP or JIS, ACS.

A.2.2 pH adjustment

Adjust the pH of the salt solution to a value of 3,5 ± 0,1 (measured at 25 °C ± 2 °C) by adding the following reagents to 10 l of the sodium chloride solution.

First add to the sodium chloride solution 12 ml of nitric acid (HNO_3, ρ = 1,42 g/ml) and 17,3 ml of sulfuric acid (H_2SO_4, ρ = 1,84 g/ml). Adjust the pH of the solution, thereafter, to the specified value of 3,5 ± 0,1 by adding the necessary quantity of a sodium hydroxide (NaOH) solution of mass fraction 10 % (approximately 317 g is required).

A.3 Test solution for simulating the corrosive effects of ocean water

A.3.1 Preparation of stock solutions

Prepare the following three stock solutions using reagent-grade chemicals and distilled or deionized water with a conductivity not higher than 2 mS/m (equal to 20 µS/cm) at 25 °C ± 2 °C.

Stock solution A

Magnesium chloride hexahydrate, $MgCl_2 \cdot 6H_2O$	3 889 g
Calcium chloride, anhydrous, $CaCl_2$	405,6 g
Strontium chloride hexahydrate, $SrCl_2 \cdot 6H_2O$	14,8 g

Dissolve the given amounts of the salts in distilled or deionized water and dilute to a total volume of 7 l. Store the solution in well-stoppered glass containers.

Stock solution B

Potassium chloride, KCl	486,2 g
Sodium hydrogen carbonate, $NaHCO_3$	140,7 g
Potassium bromide, KBr	70,4 g
Boric acid, H_3BO_3	19,0 g
Sodium fluoride, NaF	2,1 g

Dissolve the given amounts of the salts in distilled or deionized water to a total volume of 7 l. Store the solution in well-stoppered amber containers.

Stock solution C

Barium nitrate, $Ba(NO_3)_2$	0,994 g
Manganese nitrate hexahydrate, $Mn(NO_3)_2·6H_2O$	0,546 g
Copper nitrate trihydrate, $Cu(NO_3)_2·3H_2O$	0,396 g
Zinc nitrate hexahydrate, $Zn(NO_3)_2·6H_2O$	0,151 g
Lead nitrate, $Pb(NO_3)_2$	0,066 g
Silver nitrate, $AgNO_3$	0,004 9 g

Dissolve the given amounts of the salts in distilled or deionized water to a total volume of 10 l. Store the solution in well-stoppered amber containers.

When adding silver nitrate to the above solution, dissolve 0,049 g of silver nitrate in water and dilute to 1 l. Add 0,100 l of this solution to stock solution C before diluting to the final volume.

A.3.2 Preparation of substitute ocean water

Dissolve 245,34 g of sodium chloride (NaCl) and 40,94 g of anhydrous sodium sulfate (Na_2SO_4) in 8 l to 9 l of water. Add slowly, with vigorous stirring, 0,200 l of stock solution A and then 0,100 l of stock solution B. Dilute to 10 l. Adjust the pH to 8,2 with a 0,1 N sodium hydroxide (NaOH) solution.

Only a few millilitres of NaOH solution should be required.

Prepare the solution and adjust the pH immediately prior to use.

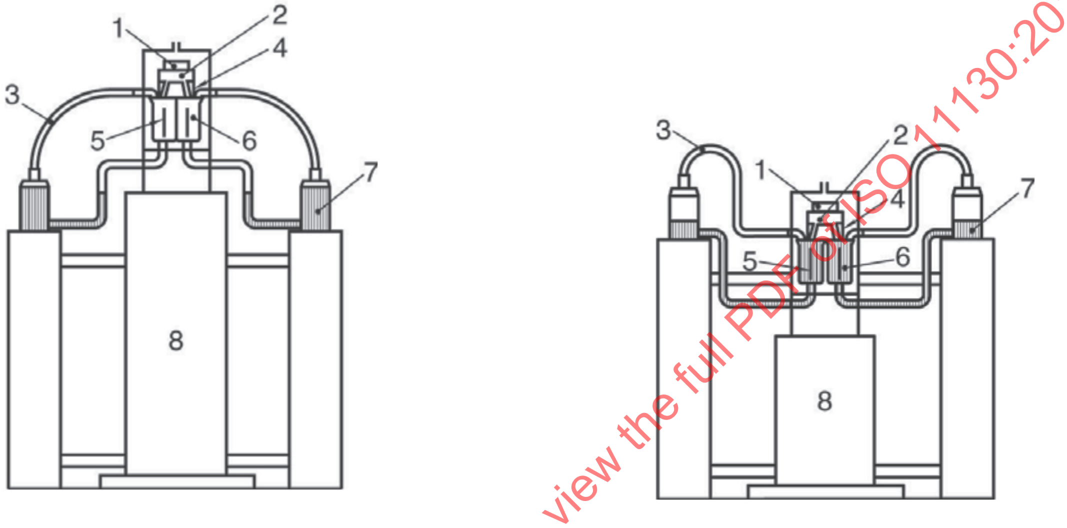
A.3.3 Preparation of substitute ocean water with heavy metals

Add 10 ml of stock solution C slowly and with vigorous stirring to 10 l of the substitute ocean water prepared as described in A.3.2.

Annex B
(informative)

Suitable apparatus for alternate immersion tests in salt solution

B.1 Example 1



a) **Dry condition**

b) **Immersion condition**

Key

1	fan	5	test specimen
2	stainless-steel duct	6	specimen cabinet
3	level control tube	7	salt solution
4	air jet	8	lift mechanism

Figure B.1 — Typical apparatus for alternative immersion tests in salt solution