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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 11130:2010(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 11130 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

This second edition cancels and replaces the first edition (ISO 11130:1999), of which Annex B has been technically revised.

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 International Standard 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 International Standard 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 International Standard 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 International Standard includes metallic materials with or without corrosion protection.

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 coating, and
- organic coatings on metals.

This International Standard is not applicable to stainless steel.

2 Normative references

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

ISO 4628-1, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 1: General introduction and designation system*

ISO 4628-2, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 2: Assessment of degree of blistering*

ISO 4628-3, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 3: Assessment of degree of rusting*

ISO 4628-4, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 4: Assessment of degree of cracking*

ISO 4628-5, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 5: Assessment of degree of flaking*

ISO 7539-1, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

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

ISO 10289, *Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests*

3 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/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.

4 Test solution

4.1 General.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized 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. Subclause 4.2 gives details of a neutral salt solution that is suitable for simulating the corrosive effect of a marine environment.

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.

4.2 Preparation.

The neutral salt solution is prepared by dissolving a sufficient mass of sodium chloride in water to give a concentration of 35 $g{\cdot}L^{-1} \pm 1 g{\cdot}L^{-1}$. The water used shall have a conductivity not higher than 2 $mS{\cdot}m^{-1}$ (equal to 20 $\mu S{\cdot}cm^{-1}$) at 25 °C ± 2 °C.

The maximum allowable impurity content in the sodium chloride solution shall be as given in Table 1:

Table 1 — Maximum impurity content in the sodium chloride solution

Impurity	Maximum permissible mass fraction %	Remarks
Copper	0,001	Determined by atomic absorption spectrophotometry or another method of similar accuracy
Nickel	0,001	
Sodium iodide	0,1	Calculated for dry salt
Total	0,5	

Prior to use, check the pH of the salt solution using electrometric measurement at 25 °C ± 2 °C or in routine checks, with a short-range pH paper which can be read in increments of 0,3 pH units or less. If the pH value determined is outside the range of 6,0 to 7,0, adjustments shall be made by adding dilute hydrochloric acid or sodium hydroxide to the salt solution.

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

5 Apparatus

5.1 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 6.1). Each test specimen shall be connected to the system using suitable insulating material.

- One or more glass or plastic containers for the reagent. Only one kind of metal, alloy or coating should be immersed in each container. Replicate specimens can share the same container.

The system should 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 is illustrated schematically in Annex B.

5.2 Materials of construction.

5.2.1 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.

5.2.2 Use of inert plastics or glass is recommended where feasible.

5.2.3 Metallic construction materials shall be selected from 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 5.2.1.

5.3 Test specimen holders.

5.3.1 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 should 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.

5.3.2 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,

- they do not retain a pool of solution in contact with the test specimen after withdrawal from the solution, and

- drainage from one test specimen does not directly come into contact with any other test specimen.

5.4 Air circulation.

5.4.1 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, but the recommendations described in 5.4.2 should be followed.

5.4.2 It is important to achieve moderate and uniform drying conditions. A mild circulation of air capable of drying specimens within about 40 min, even when coated with corrosion products and salt deposits, is recommended.

In order to achieve ideal drying conditions, the air temperature should be 27 °C ± 2 °C and the relative humidity should be 45 % ± 6 %. However, if other drying conditions are used, for example laboratory conditions, these conditions should be specified.

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 should be avoided.

6 Procedure

6.1 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 should 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.

Only one kind of metal, alloy or coating shall be immersed in the same container 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 A test duration in the range 20 days to 90 days is usually adequate for aluminium alloys and ferrous metals.

6.2 Test specimens

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

In the absence of a specified geometry, it is suggested that rectangular specimens of 90 mm × 120 mm × 1 mm be used.

A minimum of three test specimens should be used.

If the size of the test specimens is incompatible with the test apparatus, the specimens should 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 should 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 should be rinsed using clean solvent then dried.

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

6.3 Test reagent

When in the immersed position, the test specimens shall be completely covered by reagent to a minimum depth of 10 mm.

The level of reagent in the container should be maintained by the addition of distilled water to replenish evaporative losses as required.

It is recommended that the solution be changed every 168 h or when the pH varies by more than 0,3 from the original pH, whichever is sooner.

7 Calibration of the test facility

The test facility should be calibrated by tests on a standard material using test specimens with a simple geometry and uniform grain structure so that the results can be compared with published data. This calibration procedure should be repeated periodically to confirm the reproducibility of results.

8 Cleaning of test specimens

On completion of testing, test specimens should be removed from the apparatus and cleaned as thoroughly as possible in order to prevent further corrosion, by rinsing with water to remove accumulated hygroscopic salt deposit and drying (see ISO 8407).

9 Assessment of results

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

a) the appearance after the test;

b) the appearance following the removal of superficial corrosion products in accordance with ISO 8407;

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 ISO 4628;

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 (see 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.

10 Test report

10.1 The test report shall indicate the outcome of the test according to the evaluation criteria prescribed for the test. The results obtained for each test specimen should be reported and, when appropriate, the average for a group of replicate test specimens. The report may, if required, be accompanied by photographic records of the specimens.

10.2 The test report should 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 International Standard, i.e. ISO 11130:2010;

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 frequency at which the solution is changed;

i) the dimensions and geometry 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 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 specimen location permutations;

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

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

q) any abnormality or incident occurring during the entire test procedure;

r) the inspection intervals.

Annex A
(informative)

Suggested test solutions

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 demineralized 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 must 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 No. 42 Whatman filter paper[1] 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.

f) Shelf-life of the test solution:

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

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

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[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.

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^{-1} (equal to 20 µS·cm^{-1}) at 25 °C ± 2 °C to produce a concentration of 50 g·L^{-1} ± 5 g·L^{-1}. The specific gravity range for a 50 g·L^{-1} ± 5 g·L^{-1} solution is 1,029 g·cm^{-3} to 1,036 g·cm^{-3} at 25 °C.

The sodium chloride solution shall contain a mass fraction of copper less than 0,001 % and a mass fraction of nickel less than 0,001 % as determined by atomic absorption spectrophotometry or another analytical method of similar sensitivity. The mass fraction of sodium iodide shall not be higher than 0,1 % or the mass fraction of total impurities calculated for dry salt shall not be higher than 0,5 %.

If the pH of the prepared solution, measured at 25 °C ± 2 °C, is outside the range of 6,0 to 7,0, investigate the presence of undesirable impurities in the salt and/or the water.

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, $\rho = 1{,}42$ g·mL^{-1}) and 17,3 mL of sulfuric acid (H_2SO_4, $\rho = 1{,}84$ g·mL^{-1}). 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 1 % (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^{-1} 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, $Mn(NO_3)_2 \cdot 6H_2O$	0,546 g
— copper nitrate trihydrate, $Cu(NO_3)_2 \cdot 3H_2O$	0,396 g
— zinc nitrate hexahydrate, $Zn(NO_3)_2 \cdot 6H_2O$	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.

For the addition of silver nitrate to the above solution, dissolve 0,004 9 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.

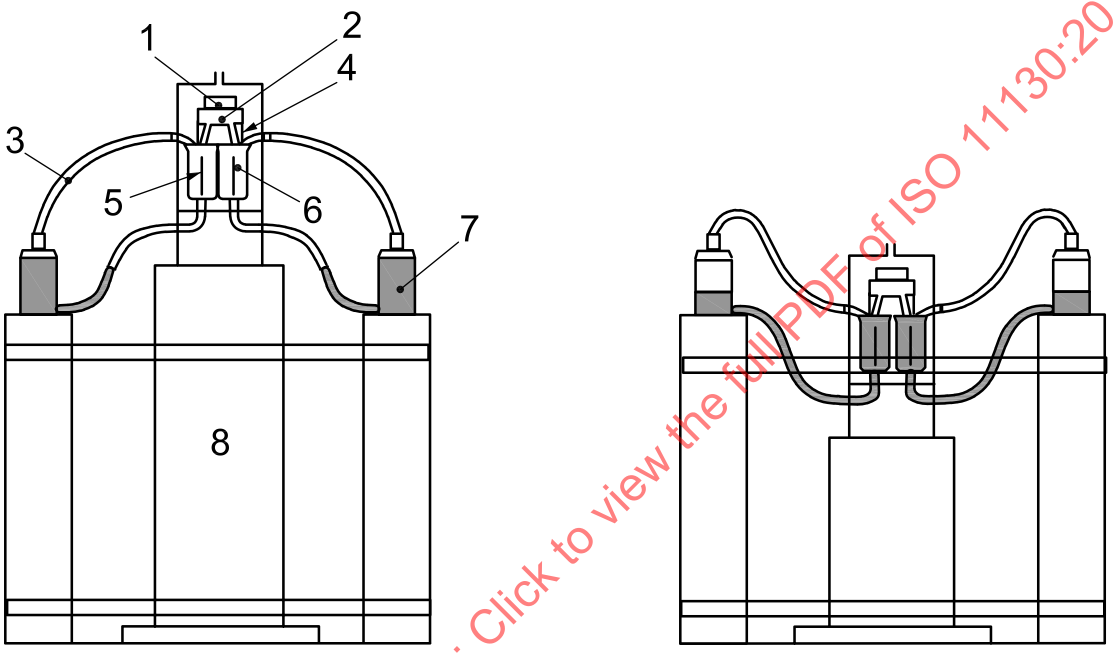
A.3.3 Preparation of substitute ocean water with heavy metals

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



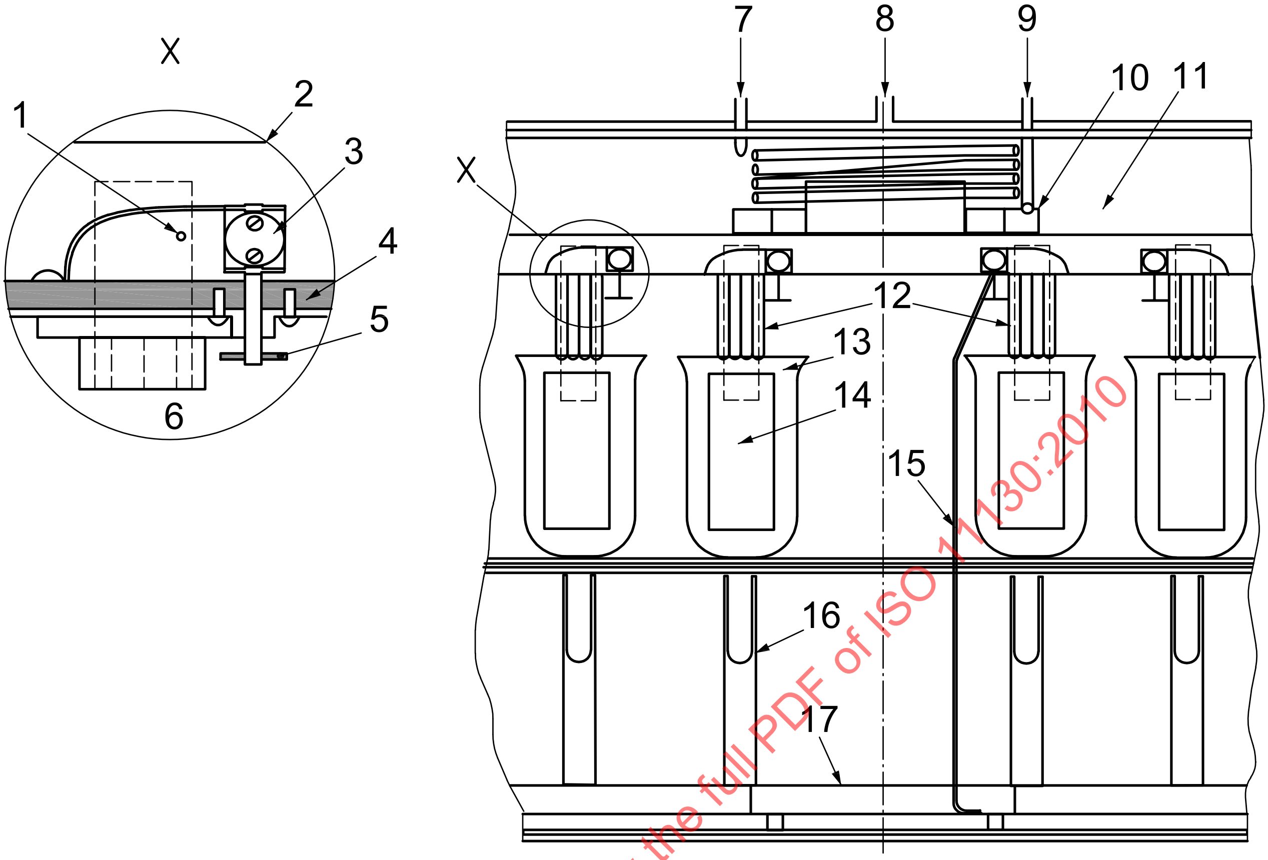
a) Dry-specimen position

b Wet-specimen position

Key

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

Figure B.1 — General layout showing unimmersed and immersed positions



Key

1	heater
2	stainless-steel duct
3	butterfly valve
4	air seat
5	adjusting key
6	air jets
7	cooling-water outlet
8	air inlet
9	cooking-water outlet
10	tray to collect condensate
11	stainless-steel duct
12	air jets
13	specimen holder
14	specimen
15	drain for condensate
16	solution inlet pipe
17	drain tray

NOTE The enlargement represents the air-flow control unit.

Figure B.2 — Detailed view of central portion of cabinet