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ISO 10270

Second edition
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Corrosion of metals and alloys — Aqueous corrosion testing of zirconium alloys for use in nuclear power reactors

Corrosion des métaux et alliages — Essais de corrosion aqueuse des alliages de zirconium utilisés dans les réacteurs nucléaires

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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.

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 are 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 is not 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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10270:1995), which has been technically revised. It also incorporates the Technical Corrigendum ISO 10270:1995/Cor 1:1997. The main changes compared with the previous edition are as follows:

- the references have been updated;
- in Clause 4, two sentences have been added: “The tests in water is performed at 18,6 MPa. The pressure is also determined by the contractor's requirements.”;
- in 5.2, “the test” has been replaced by “the measurement to avoid erroneous results from differing corrosion behaviour of the materials”;
- in 7.1, “welding grade” has been replaced by “of purity 999,9 ml/l or higher”,
- in 11.1, d), “1 day” has been replaced by “24 h”; and “at 18,6 MPa” has been added after “at 360 °C in water”.
- in 12.1, a sentence has been added: “Post-heat treatment, the coupons are inspected for any residual oxide and prepared (e.g. etched) in line with standard coupons”;
- in 12.3.4, a sentence has been added: “However, for the product acceptance test, it is better to keep the control coupons as evidence of test effectiveness.”;
- in 13.3.1, “±3 °C for steam tests, and ±6 °C for water tests” has been replaced by “±3 °C for steam tests and water tests”;
- in 13.3.3, “3 days or 14 days” has been replaced by “72 h or 336 h”;

- in 14.2, a sentence has been added: “Mass gain measurements are taken in triplicate for each specimen and a mean value calculated”;
- in 14.3.1, a sentence has been added: “The separators are such as not to induce Galvanic interaction between the samples and the separators”; “zirconium or zirconium alloy” has been added before “stainless steel”;
- in Clause 16, “including its number and year of publication” has been added before “this document”;
- in A.3.1, “3 % (m/m) of hydrofluoric acid (7.7), 39 % (m/m) of nitric acid (7.8)” has been replaced by “40 g/kg ± 20 g/kg of hydrofluoric acid (7.7), 450 g/kg ± 50 g/kg of nitric acid (7.8)”, and a sentence has been added: “A different acid ratio may be chosen to satisfy the requirement of a smooth and shining surface given in 13.4.1;
- in A.3.2, “9 % (m/m) ± 1 % (m/m)” has been replaced by “90 g/kg ± 10 g/kg” and “30 % (m/m) ± 5 % (m/m)” has been replaced by “300 g/kg ± 50 g/kg”;
- in A.4.2,“25 % (V/V)” has been replaced by “250 ml/l” and “50 % (V/V)” has been replaced by “500 ml/l”;
- in Clause A.6, the following text has been added: “or placed into the oven at a recommended temperature of 60 °C to 70 °C for 1 h" and “The specimens need to be cooled to room temperature after removing from the oven”.

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

Corrosion of metals and alloys — Aqueous corrosion testing of zirconium alloys for use in nuclear power reactors

WARNING — This document can involve the use of hazardous materials, operations and equipment (see Clause 9). It is the responsibility of the user of this document to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies:

a) the determination of mass gain;

b) the surface inspection of products of zirconium and its alloys when corrosion is tested in water at 360 °C or in steam at or above 400 °C;

c) the performance of tests in steam at 10,3 MPa.

This document is applicable to wrought products, castings, powder metallurgy products and weld metals.

This method has been widely used in the development of new alloys, heat-treating practices and for the evaluation of welding techniques. It is applicable for use in its entirety to the extent specified for a product acceptance test, rather than merely a means of assessing performance in service.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological 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/

3.1
etching
process for the removal of surface metal by action of acids in water

3.2
control coupon
zirconium alloy specimen of known performance used to monitor the validity of the test

3.3
high mass gain coupon
zirconium alloy specimen that has been specially heat-treated to produce a mass gain higher than the maximum given in materials acceptance specifications and which is used for verifying the severity of the test procedure

4 Principle

Specimens of zirconium or its alloys are exposed to high-pressure water or steam at elevated temperatures for 72 h or 336 h. The corrosion is normally measured by the gain in mass of the specimens and by the appearance of an oxide film on the specimen surfaces. In some instances, such as weld evaluation, mass gain measurements are either impractical to make or are not required. When so specified, the appearance of the specimen is the sole criterion for acceptance. The test pressure in steam is 10,3 MPa. The tests in water is performed at 18,6 MPa. The pressure is also determined by the contractor's requirements.

5 Significance

5.1 Specimens are normally tested after careful etching and rinsing. Specimens with as-manufactured surfaces may also be tested without further surface removal.

5.2 When tubing with a second material clad on the surface is to be tested, the internal cladding is removed prior to the measurement to avoid erroneous results from differing corrosion behaviour of the materials.

6 Interference

Autoclave loads that have one or more specimens showing gross oxidation can, by contamination of the environment, affect results of other specimens in the autoclave.

7 Reagents and materials

During the test, unless otherwise stated, only reagents of recognized analytical grade and only water as described in 7.4 or 7.5 are used.

7.1 **Argon gas**, of purity 999,9 ml/l or higher.

7.2 **Nitrogen gas,** for purging or controlling oxygen content.

7.3 **Argon-hydrogen mixture,** for purging to remove dissolved oxygen.

7.4 **Grade A water,** purified water having an electrical resistivity of not less than 1 MΩ·cm at 25 °C as measured before the start of the test.

7.5 **Grade B water,** deionized or demineralized water having an electrical resistivity of not less than 0,5 MΩ·cm at 25 °C. Grade A water can be used instead of grade B water.

7.6 **Detergents and solvents,** for specimen cleaning, including ethanol and acetone.

7.7 **Concentrated hydrofluoric acid** (HF).

7.8 **Concentrated nitric acid** (HNO_3).

7.9 **Concentrated sulfuric acid** (H_2SO_4).

7.10 **Control coupon**.

7.11 **High mass gain coupon**.

8 Apparatus

The apparatus consists of equipment for:

a) etching the specimen when required;

b) measuring the specimens' surface area and mass, the water resistivity and pH, the test temperature and pressure, and the etch and rinse temperature;

c) performing the water or steam corrosion tests at elevated temperatures and pressures.

8.1 Etching equipment, comprising an acid bath, a running water rinse and a deionized water rinse needed for proper metal removal and stain-free rinsing. Polyethylene or polypropylene tanks are commonly used with a bottom feed for running water rinses. Specimen hangers are generally made of type 300 series stainless steel. When many specimens are processed, a mechanical dipper for the etching process is useful.

8.2 Autoclaves, constructed of type 300 series stainless steel or nickel base alloys such as UNS grade N06600 or N066901. The autoclave is fitted with devices for measurement and control of pressure and temperature, safety devices and venting valves. Control systems for pressure and temperature are adequate to meet the requirements of this document. Sample holders and other internal accessories are also constructed of type 300 or type 400 series stainless steel, or nickel-base alloys such as UNS[1] grade N06600 or N06690.

8.3 Specimen size measuring equipment, accurate to 50 µm.

8.4 Analytical balance, accurate to 0,1 mg.

9 Hazards

9.1 The chemicals used in preparing specimens for this test are hazardous. Detailed information on the safe handling of organic compounds, acids and products of zirconium and its alloys are obtained from competent sources.

9.2 High-temperature, high-pressure autoclave operation is in accordance with the manufacturer's instructions.

9.3 Hydrogen gas used for the addition to the autoclave steam supply is handled in accordance with the guidelines for explosives and inflammable substances.

9.4 Cold water is not added directly to the autoclave vessel in order to accelerate cooling upon the completion of testing.

10 Sampling, test specimens and test units

10.1 The size and the quantity of the test specimens, the method of selection, surface preparation and test acceptance criteria are specified in the product specification or by agreement between the purchaser and the seller as stated in the purchase contract. The total surface area for each specimen is a minimum of 10 cm^2.

10.2 Each specimen and control coupon is individually identified.

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1) UNS grades N06600 and N06690 are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

11 Preparation of apparatus

11.1 General requirements for new or reworked autoclaves (8.2) and parts of autoclaves previously used for testing materials other than in accordance with this document are as follows.

a) Before specimens are tested in a new or reworked autoclave, or in one having new valves, tubing, gaskets, etc. which are in contact with the test specimen, clean the apparatus thoroughly, wipe with ethanol or acetone (7.6) and rinse twice with grade B water (7.5).

b) Dry the autoclave or auxiliary equipment by vacuum cleaning, or drain and wipe with a clean, lint-free cloth and inspect carefully to ensure freedom from contamination. There is no visible contamination, such as lubricant, residues, dust or dirt, loose oxides or rust, or an oil or grease film on the water surface, internal surface, gasket or head surfaces.

c) Clean all new and reworked fixtures and jigs to be used in the autoclave and rinse in hot grade B water.

d) Autoclave the fixtures and jigs for at least 24 h at 400 °C in steam at 10,3 MPa or, preferably, at 360 °C in water at 18,6 MPa.

e) Inspect the parts for corrosion products. If corrosion products are found or electrical resistivity of the residual water after the test measures less than 0,1 MΩ·cm, clean and autoclave the parts again.

11.2 General requirements for autoclaves and parts in continuous use for corrosion testing in accordance with this document are as follows.

a) All autoclaves, fixtures, parts and jigs that have been in continuous use and have shown satisfactory behaviour in prior tests are rinsed with grade B water.

b) The fixtures and jigs for corrosion products are inspected after each test and items showing loose corrosion products are re-worked and re-prepared.

12 Calibration and standardization

12.1 High mass gain coupon preparation

High mass gain coupons (7.11) is selected from a previously tested lot. For Zircaloy type alloys, the selected material is specially heat-treated prior to exposure in order to produce the desired mass gain in the autoclave test. Heating for 8 h at 900 °C and cooling to 300 °C at a rate not exceeding 3,3 °C/min in an inert atmosphere [e.g. Ar (7.1)] or vacuum is a suggested procedure, but alternative procedures can be used. This treatment is used in the case of Zircaloy type alloys. Other alloy types can require different heat-treatment. Post-heat treatment, the coupons are inspected for any residual oxide and prepared (e.g. etched) in line with standard coupons.

12.2 Autoclaves

12.2.1 Prior to use for product acceptance testing, an autoclave is profiled thermally as given in 12.4.2 and demonstrates acceptability by testing at least three control coupons: one each at the top, middle and bottom of a useful volume. The test results are incorporated in the certification document for the autoclave acceptance test. When desired, high mass gain coupons may also be used.

The new or used autoclave is considered acceptable if each control coupon mass gain is reproducible within the previously established control coupon mean mass gain, ±3 standard deviations.

12.2.2 The control coupon lot (7.10) and, when desired, the high mass gain coupon lot (7.11) mass gain mean and standard deviation are established by a minimum of one autoclave test as follows.

a) Randomly select 12 specimens from either the control coupon lot or the high mass gain coupon lot or both.

b) Prepare all specimens in accordance with 13.4 in order to meet the pre-test requirements of this document.

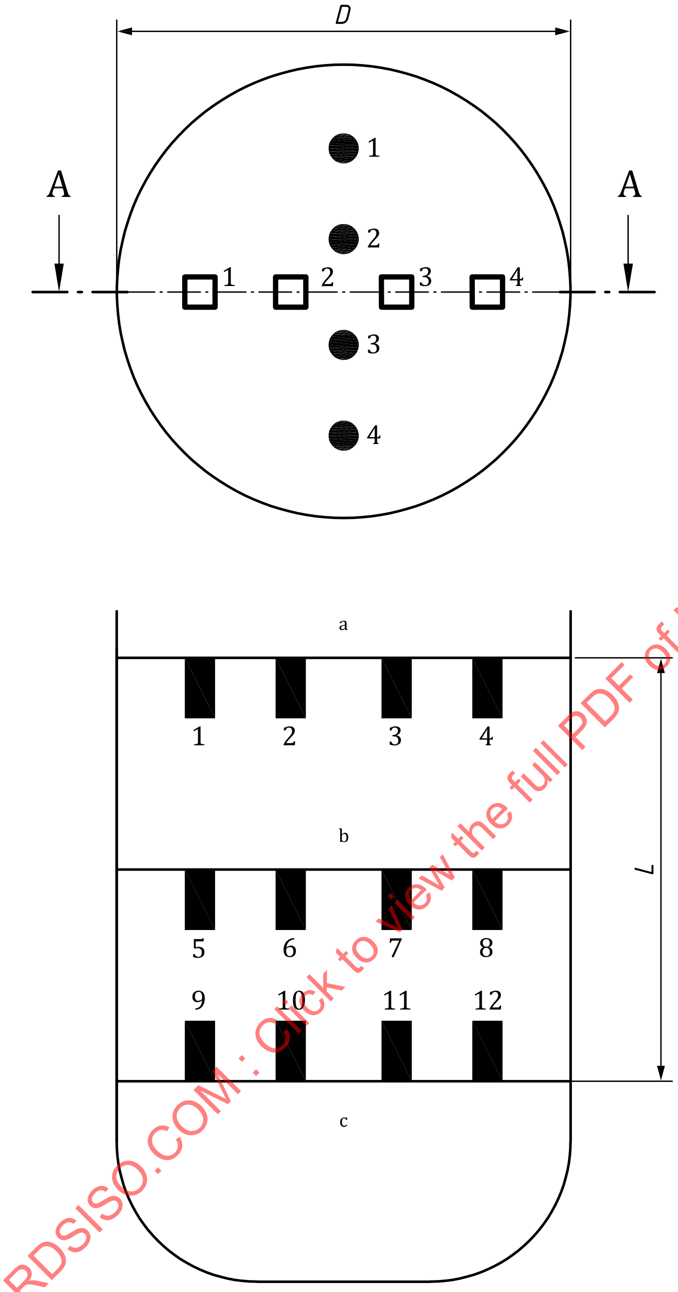
c) Locate the 12 or 24 specimens in a fixture or jig (see Figure 1) and place the fixture or jig inside the useful volume of the autoclave.

d) Complete the steam or water corrosion test in accordance with any one of the four methods in 14.3.

e) Remove the specimens and weigh in accordance with the requirement of this document.

f) Calculate and establish the mass gain mean and standard deviation [$(n - 1)$ method] of each set of coupons for the test method used.

For the product acceptance tests, the mean value and standard deviation for the control coupons is the value established as described above or it is calculated periodically using all accepted values determined over the preceding three-month period but not less than 21 values.



Key

█	sample
●	control coupons
☐	high mass gain coupons
a	Top.
b	Middle.
c	Bottom.
A	axis
D	internal diameter of useful autoclave
L	length of useful autoclave

Figure 1 — Control coupon and high mass gain coupon positioning for establishing mass gain mean and standard deviation

12.2.3 An alternative method for establishing the mass gain mean and standard deviation is as follows.

a) Expose the control coupons to be used in three different tests. In each test, a minimum of one specimen is exposed in each of the top, middle and bottom positions of the autoclave.

b) Determine the mass gain.

c) The mean value for control coupons is the mean for the three tests and three positions.

d) The standard deviation for the control coupon lot is calculated by the (n − 1) method using the data from all of the control coupons taken from the same material lot.

12.3 Use of control coupons

12.3.1 Each autoclave run used for acceptance of product contains at least three control coupons with one at the top, one at the middle and one at the bottom of the specimen load.

12.3.2 The control coupons may be as-manufactured or etched before testing, but if etched, the surfaces exhibit no stains, pits or areas of abnormal etching attack.

12.3.3 An autoclave test is considered acceptable only if each post-test control coupon mass gain is not less than the established mean value −3 standard deviations and the visual appearance of each control coupon is equal to or better than the product acceptance standard. If a control coupon post-test mass gain exceeds the mean value +3 standard deviations, or the specified mass gain value and one or more test specimens from the corresponding location in the autoclave fail to meet the mass gain acceptance criterion, the failed specimen(s) may be discarded and a new test made to determine conformity.

12.3.4 Control coupons may be re-used after mechanical removal of oxide film followed by etching as described in 13.4.1. However, for the product acceptance test, it is better to keep the control coupons as evidence of test effectiveness.

12.4 Calibration

12.4.1 The temperature measurement and recording systems used to determine conformity are calibrated at least every six months and do not deviate more than ±3 °C from the calibration reference materials traceable to national standards.

12.4.2 Vertical thermal profiles of the autoclaves at the test temperatures are made at least once in each six-month period, or whenever the heaters or the control thermocouples are adjusted or replaced. The axial extent of the autoclave used for performing the product acceptance testing is restricted to the volume shown to be within ±3 °C of the recorded autoclave temperature, after temperature compensation for calibration of the thermocouples. This volume is considered to be the useful volume. The profile thermocouples may be located at the centre or near the radial extremity of the autoclave volume.

12.4.3 Pressure-measuring devices are calibrated annually, and the recorded reading is within ±0,35 MPa of the calibrated device over the range used for testing.

13 Conditioning

13.1 Test water quality

The water used to conduct the corrosion test is grade A water (7.4) and has an oxygen content not exceeding 45 pg/g. The oxygen content specification may be met either by direct measurement using the appropriate method specified in ISO 5813 or ISO 5814 or by the use of venting method A or method B specified in 14.3.

13.2 Autoclave load restrictions

The surface area of specimens loaded in a static autoclave does not exceed 0,1 m^2/l of autoclave volume.

13.3 Test conditions

13.3.1 Temperature

The recorded temperature within the volume used for testing is the specified value ±3 °C for steam tests and water tests.

13.3.2 Pressure

The recorded pressure is as specified ±0,7 MPa for steam tests and ±1,4 MPa for water tests.

13.3.3 Time

The recommended exposure times are 72 h or 336 h. The exposure time tolerance at the specified temperature and pressure is $^{+8}_{\ 0}$ h. The time at specified conditions does not need to be continuous.

13.3.4 Tests

Tests where temperature or pressure limits or both are exceeded for not more than 10 % of the nominal test time, but where control coupons indicate satisfactory behaviour may, at the discretion of the test laboratory, be deemed acceptable.

13.4 Specimen preparation

13.4.1 Etched specimens

Specimens are thoroughly cleaned prior to acid etching and carefully rinsed to prepare the surfaces for testing in a manner that eliminates the effects of machining, grinding or other techniques used to obtain a specimen of the desired site. Any etching technique that produces a desired smooth and shiny finish free of stains may be used. Zirconium-niobium alloys etch to a matt finish. An example of a suitable procedure for etching and rinsing is given in Annex A.

13.4.2 As-manufacturer specimens

These specimens are thoroughly cleaned prior to testing to avoid contamination of the autoclave which could, in turn, adversely affect other specimens used in the test. The extent of specimen cleaning is specified by agreement between contracting parties. An example of a suitable cleaning procedure is given in Annex A.

14 Procedure

14.1 Inspection of specimens

Examine the specimen for folds, cracks, blisters, foreign material, lustre, brown acid stains, etc. Discard or re-prepare any etched specimen exhibiting acid stains or dull surfaces.

14.2 Dimensions, weight and inspection

Measure each test specimen, either before or after testing, to ±50 µm and calculate the surface area rounded off to the nearest 1 mm^2. Weigh the specimens to the nearest 0,1 mg with the analytical balance (8.4). Check the balance with a calibrated mass in the appropriate range before use and zero it after each fourth weighing. Weigh the specimens when they are thoroughly dry and at the same temperature as the balance. Mass gain measurements are taken in triplicate for each specimen and a mean value calculated.

14.3 Autoclaving

14.3.1 Placing of test specimens

Place the clean and weighed test specimens on the clean fixtures in a manner which precludes specimen-to-specimen contact. The separators are such as not to induce Galvanic interaction between the samples and the separators. Autoclaved (8.2) zirconium or zirconium alloy, stainless steel type 300 or type 400 series washers or wire-mesh grids may be used as separators. Immediately before immersing in the autoclave (8.2), the fixtures containing the specimens may be rinsed in grade B water (7.5). Immediately before the operation, rinse the autoclave twice with grade B water. Place the specimens and fixtures in the useful volume of the cleaned and rinsed autoclave.

14.3.2 Venting method A

14.3.2.1 Water tests

It is necessary to determine the volume of water required to ensure that the parts being tested are covered with water during the entire test period. The means of achieving this is as follows.

First, pre-determine the required water level by filling the autoclave with sufficient water to cover the samples. Measure the volume of water added, V_1. Calculate the volume of water, V_2, to be added so that the specimens are completely immersed in the liquid phase at the test temperature, using Formula (1):

$$V_2 = \frac{V_1}{v'} + \frac{V_3 - V_1}{v''} \tag{1}$$

where

V_3 is the free volume of autoclave (volume of autoclave minus volume of samples), best measured by filling the autoclave containing samples to the brim with a measured volume of water;

v' is the specific volume of water at the test temperature, given in Table 1;

v'' is the specific volume of steam at the test temperature, given in Table 1.

Values for other temperatures are given in Reference [3].

Table 1 — Relationship between temperature and the specific volumes of water

Temperature °C	Volume v'	 v''
146	1,086 2	434,7
260	1,275 5	42,15
288	1,358 5	26,84
316	1,477	16,55
343	1,667	10,17
360	1,894	6,943

Clean and thoroughly rinse the autoclave before use. Add grade A water to a 10 % excess above the calculated volume, V_2. Activate the autoclave heating units after the autoclave has been attached and sealed. When the internal temperature reaches about 150 °C, open the vent valve to the atmosphere or the venting manifold and collect the condensate to remove the 10 % excess water previously added. Close the valve or valve manifold, then control the autoclave for test requirements of temperature and pressure.

14.3.2.2 Steam tests

Place the fixtures and specimens in a clean, thoroughly rinsed autoclave. Add grade A water (7.4) until the autoclave is at least one quarter to three quarters full. Activate the autoclave heating units after the autoclave has been sealed. When the internal temperature exceeds 110 °C, open the vent valve to the atmosphere or the venting manifold for sufficient time for degassing to be complete, and close the valve. As the temperature and pressure continue to rise, open and close the vent valve, as necessary, to maintain the required pressure.

14.3.3 Venting method B

14.3.3.1 Water tests

Load the clean, thoroughly rinsed autoclave with fixtures and specimens. Fill with grade A water as in venting method A (see 14.3.2), or if the autoclave is equipped with an automatic venting system, fill with enough water to cover the specimens. Seal the autoclave and activate the heating units. Vent the autoclave for at least 6 min after 90 °C is reached, then control the autoclave for test requirements of temperature and pressure.

14.3.3.2 Steam tests

Load the autoclave with fixtures, specimens and grade A water as in venting method A (see 14.3.2). Seal the autoclave and activate the heating units. The vent valve is open a minimum of 10 min prior to reaching 150 °C. As the temperature and pressure continue to rise, maintain the pressure at test requirements by momentarily opening the vent valve.

14.3.4 Closed system method C

14.3.4.1 Water tests

Prepare degassed grade A water and store in a separate closed system. Place the fixtures and specimens in the clean, dry autoclave and assemble except for the filling connection. Evacuate the autoclave to approximately 1,3 kPa pressure, backfill with argon (7.1) and re-evacuate. Backfill the autoclave with argon to a gauge pressure of 34 kPa and add the required amount of water without the 10 % excess of venting method A (see 14.3.2) to the autoclave through a closed system, then seal the autoclave and activate the heating units. No venting is required.

14.3.4.2 Steam tests

Use a procedure similar to that described in 14.3.4.1 to fill the autoclave (one quarter to three quarters). After the heating units are activated, vent to attain the required test pressure. Fill the autoclave in a closed system using degassed water. Test the grade A water for pH, electrical resistivity and oxygen content immediately before filling, then record the results.

The stated values of pH and electrical resistivity shall be met after the measured values are corrected to 25 °C.

14.3.5 Refreshed autoclaving, method D

14.3.5.1 General

A refreshed autoclave with a high-pressure, constant-volume pump, a preheater with controller and a separate over-temperature device, suitable back-pressure controller and system over-pressure protection is required. Operate the pump at rates that permit proper functioning of the pressure control system and maintain the pH within 0,2 units and the electrical resistivity of the effluent water at 0,4 MΩ·cm or more. Introduce the feed water, which is initially degassed to less than 45 pg/g oxygen at the bottom of the autoclave and bleed the effluent from the top.

14.3.5.2 Water tests

Place the samples in clean autoclaves nearly full of grade A water. Close the autoclave cover and connect the necessary piping and instrument lines. Pump additional grade A water into the autoclaves until no air bubbles come out of the open bleed-off valve. Close the bleed-off valve and set the controller to the operating pressure. When the autoclave is pressurized and the effluent water meets the resistivity, pH and dissolved oxygen requirements, turn on the heaters and bring the vessel to operating temperature. When the test is completed, turn off the heaters, disconnect the feed water and cool the autoclave to less than 100 °C before opening.

14.3.5.3 Steam tests

Follow the procedure for the water test (see 14.3.5.2). Autoclave blow-down through a bottom connection or dip tube is permitted if post-test water samples are not required.

14.4 Post-test measurements and inspection

Carefully remove the specimen from the fixtures, using clean gloves or forceps to prevent scratches. The specimens may be rinsed in grade B water (7.5) or ethanol (7.6) and dried. Store the specimens in a clean, dry container at the same temperature as the analytical balance (8.5) for at least 1 h before weighing. Weigh the specimens and measure the dimensions, if needed. The specimens are examined in accordance with 15.2.

15 Calculation or interpretation of results

15.1 Calculation of mass gain

Calculate and record the increase in specimen mass per unit area using Formula (2):

$$\Delta m = \frac{(m_2 - m_1)}{A} \tag{2}$$

where

Δm is the mass gain per unit area;

m_1 is the pre-test mass of the specimen;

m_2 is the post-test mass of the specimen;

A is the total surface area of the specimen.

NOTE Generally, throughout the industry the mass gain is reported in milligrams per square decimetre (mg/dm^2).

15.2 Visual interpretation of surface appearance

After testing, examine each specimen for colour, lustre, surface irregularities and corrosion products. Compare against visual standards and record the results. The visual examination is performed in a light environment as agreed upon between the purchaser and the testing laboratory.

15.3 Invalid tests

Any test not meeting the parameters of 12.3.3, 13.1, 13.2 and 13.3 or where the test operator can define a condition that is significantly different from that normally observed may be declared invalid and the test repeated. The repeated test and the reason for it are cited in the test report (see Clause 16).

16 Test report

The test report includes the following information:

a) a reference to this document (including its number and year of publication, i.e. ISO 10270:2022);

b) the laboratory where testing was performed;

c) the autoclave number and test date;

d) a description of the tested material (type of alloy, chemical composition, lot number, heat-treatment, surface finish, etc.);

e) the electrical resistivity and pH value (if measured) of the water before test;

f) the test temperature, pressure, time, type of test and venting method;

g) the mass gain, when required, and remarks on the visual appearance of each specimen and control coupon;

h) reference to criteria for the visual appearance of specimens and control coupons;

i) any deviations from the procedure;

j) any unusual features observed.

Annex A
(informative)

Guide to specimen preparation

A.1 Tubes with a second material clad on inner diameter

When it is necessary for corrosion test tubes, the inner surface cladding is completely removed to avoid erroneous results due to differences in corrosion rates for the two materials.

A.2 Cleaning

Clean the specimens with chemical detergents or organic solvents (7.6). A non-metallic brush may be used if required. If solvents are used, the specimens may be cleaned by wiping or total immersion. Immediately after detergent cleaning, thoroughly rinse the test specimens in hot (about 50 °C) flowing water for at least 5 min. After cleaning, handle all specimens with clean, lint-free gloves. Clean the surfaces of all tools, fixtures and the like that come into contact with the clean specimens in a manner equivalent to that used for the specimens. Store all specimens so as to maintain cleanliness.

A.3 Etching

A.3.1 The approximate bath composition for unalloyed zirconium and the zirconium-tin alloy is 40 g/kg ± 20 g/kg of hydrofluoric acid (7.7), 450 g/kg ± 50 g/kg of nitric acid (7.8), and the remainder distilled or demineralized water. Other concentrations of reagents may be used provided that the equivalent final concentrations are obtained. The etching bath temperature does not exceed 50 °C. A different acid ratio may be chosen to satisfy the requirement of a smooth and shining surface given in 13.4.1.

A.3.2 For zirconium-niobium alloys, the bath composition is 90 g/kg ± 10 g/kg of hydrofluoric acid, 300 g/kg ± 50 g/kg of nitric, 300 g/kg ± 50 g/kg of sulfuric acid (7.9), and the remainder distilled or deionized water. This bath is controlled between 50 °C and 60 °C.

A.3.3 Generally, 50 μm to 100 μm of the surface of each coupon is removed by etching. Since the rate of metal dissolution is a function of both temperature and acid concentration, the etching rate is determined with a special test coupon before actual test specimens are etched. The etching rate is checked periodically when a large number of specimens are etched. For the zirconium-tin alloys, the batch of etching solution is discarded when the etching rate is less than 25 μm/min per surface or when a total of 425 cubic centimetre of the surface area per litre of acid has been etched.

A.3.4 When etching zirconium-niobium alloys, it is necessary to limit the area of specimens etched at one time to 30 cm^2 per litre of solution to get a good surface finish, but the bath does not need to be discarded each time.

A.3.5 Freshly etched zirconium alloy surfaces are bright and lustrous, and the bath does not cause preferential attack, except at the top edge or around the holes and identification marks. If preferential etching does occur elsewhere, the test specimen is discarded or abraded and re-etched. If staining does occur, the specimen is re-etched.