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Corrosion of metals and alloys — Accelerated testing involving cyclic exposure to salt mist, “dry” and “wet” conditions

Corrosion des métaux et alliages — Essais accélérés comprenant des expositions cycliques à des conditions de brouillard salin, de séchage et d'humidité



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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14993 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Annexes A and B of this International Standard are for information only.

Introduction

Corrosion of metallic materials with or without corrosion protection is influenced by many environmental factors, the importance of which may vary depending on the type of metallic material and depending on the type of environment. It is impossible, therefore, to design accelerated laboratory corrosion tests in such a way that all environmental factors influencing the resistance to corrosion are taken into account. Laboratory tests are therefore designed to simulate the effects of the most important factors enhancing the corrosion of metallic materials.

The accelerated corrosion test method described in this International Standard is designed to simulate and enhance the environmental influence on a metallic material of exposure to an outdoor climate where exposure to salt-contaminated conditions occurs and may promote corrosion. The test method involves cyclic exposure of test specimens to a mist of salt solution, to drying conditions and to periods of high humidity. However, the method is mainly intended for comparative testing and the results obtained do not permit far-reaching conclusions on the corrosion resistance of the tested metallic material under the whole range of environmental conditions within which it may be used. Nevertheless, the method provides valuable information on the relative performance of materials exposed to salt-contaminated environments similar to those used in the test.

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Corrosion of metals and alloys — Accelerated testing involving cyclic exposure to salt mist, “dry” and “wet” conditions

WARNING — This International Standard may involve hazardous materials, operations and equipment. It does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies the apparatus and test procedure to be used in conducting accelerated corrosion tests for the comparative evaluation of metallic materials with or without permanent corrosion protection or temporary corrosion protection in salt-contaminated outdoor environments. The test involves cyclic exposure of the specimens to neutral salt mist, “dry” and “wet” conditions. The type of test specimen and the exposure period are not specified.

The particular advantages of this test over conventional accelerated tests such as the neutral salt spray test (NSS) lie in its ability to better reproduce the corrosion that occurs in outdoor salt-contaminated environments.

The accelerated corrosion tests involving cyclic exposure to salt mist, “dry” and “wet” conditions apply to:

- metals and their alloys;
- metallic coatings (anodic and cathodic);
- conversion coatings;
- anodic oxide coatings;
- organic coatings on metallic materials.

NOTE Methods of test for coatings to determine their resistance, in the presence of scribe marks through to the substrate, to various cyclic corrosion conditions which include the condensation of water on the test panels during periods of humidity are given in ISO 11997-1:1998.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 8407:1991, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

3 Test solution

3.1 General

Given in 3.2 and 3.3 are instructions for the preparation and use of a neutral 5 % sodium chloride solution.

3.2 Preparation of the sodium chloride solution

Dissolve a sufficient mass of sodium chloride in distilled or deionized water with a conductivity no higher than $20 \mu\text{S}/\text{cm}$ at $25 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ to produce a concentration of $50 \text{ g/l} \pm 5 \text{ g/l}$. The sodium chloride concentration of the sprayed solution collected shall be $50 \text{ g/l} \pm 5 \text{ g/l}$. The specific gravity range for a $50 \text{ g/l} \pm 5 \text{ g/l}$ solution is 1,029 to 1,036 at $25 \text{ }^\circ\text{C}$.

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

NOTE If the pH of the prepared solution, measured at $25 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$, is outside the range 6,0 to 7,0, the presence of undesirable impurities in the salt and/or the water should be investigated.

3.3 pH adjustment

Check the pH of the salt solution by using electrometric measurement at $25 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ or in routine checks, with a short range pH paper that can be read in increments of 0,3 pH units or less. Adjust the pH of the salt solution on the basis of the pH of the sprayed solution so that the pH of the sprayed solution collected in each of the collection devices is within the range 6,5 to 7,2 at $25 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$. Adjustments of the pH of the salt solution are to be made by adding to the salt solution diluted hydrochloric acid or sodium hydroxide of analytical grade. The sodium chloride concentration of the solution collected in each of the collection devices shall be $50 \text{ g/l} \pm 5 \text{ g/l}$.

NOTE Changes in pH may result from loss of carbon dioxide from the solution when it is sprayed. Such changes may be avoided by reducing the carbon dioxide content of the solution by, e.g., heating it to a temperature greater than $35 \text{ }^\circ\text{C}$ before it is placed in the apparatus, or by making the solution from freshly boiled water.

4 Apparatus

All components in contact with the salt mist spray or the test solution shall be made of, or lined with, materials resistant to corrosion by the test solution and which do not influence the corrosivity of the sprayed test solution. The apparatus shall include the following components.

4.1 Exposure equipment

4.1.1 Exposure cabinet, with a capacity of not less than $0,4 \text{ m}^3$. For large-capacity cabinets, it is necessary to ensure that during periods of exposure to salt mist spray, the necessary conditions of homogeneity and distribution of spray are met. The upper parts of the cabinet shall be designed so that drops of sprayed solution formed on its surface do not fall on the specimens being tested.

The size and shape of the cabinet shall be such that during periods of exposure to salt mist spray, the collection rate of solution in the cabinet is within the limits specified in 7.2.

NOTE A schematic diagram of one possible design of exposure cabinet and associated apparatus for salt mist cyclic corrosion testing is shown in annex A.

4.1.2 Humidity and temperature control system, that maintains the cabinet and its contents at the specified temperature and humidity (see 7.1). The temperature shall be measured at a position at least 100 mm from the walls of the cabinet.

4.1.3 Spraying device, for spraying the salt solution during periods of exposure to salt mist spray comprising a supply of air at controlled pressure, a reservoir containing the solution to be sprayed and one or more atomizers.

The compressed air supplied to the atomizers shall be passed through a filter to remove all traces of oil or solid matter and shall be at an absolute pressure of 70 kPa to 170 kPa.

4.1.4 Air saturator, consisting of a saturation tower containing hot water at a temperature several degrees Celsius higher than that of the cabinet. In order to prevent evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer, by passage through this tower.

During periods of exposure to the salt mist, the appropriate temperature depends on the pressure used and on the type of atomizer nozzle and shall be adjusted so that the rate of collection of spray in the cabinet and the concentration of the collected spray are kept to within the specified limits (see 7.2). The level of water must be maintained automatically to ensure adequate humidification.

The atomizers shall be made of inert material, e.g. glass or plastic materials. Baffles may be used to prevent direct impact of spray on the test specimens and the use of adjustable baffles is helpful in obtaining constant distribution of spray within the cabinet. The level of the salt solution in the supply tank shall be maintained automatically to ensure constant spray delivery throughout the test.

4.1.5 Collecting devices, at least two, consisting of clean funnels with a collecting area of approximately 80 cm² made of glass or other chemically inert material, with the stems inserted into graduated cylinders or other similar containers. The purpose of the collecting devices is to confirm that the collection rate is within specified limits (see 7.2). They shall be placed in the zone of the cabinet where the test specimens are placed, one close to the inlet of spray and one remote from an inlet and in such a way that only spray, and not liquid falling from specimens or from parts of the cabinet, is collected.

4.1.6 Air dryer, comprising a heating device and fan to supply dried air of the specified humidity during "dry" periods (see Table 1).

4.1.7 Exhaust system, by which air is exhausted from the spray cabinet. It shall not be influenced by the back pressure of the atmosphere when the air is released at a point outside the building.

5 Test specimens

5.1 The number and type of test specimens, their shape and their dimensions shall be selected according to the specification for the material or product being tested. When not so specified, these details shall be mutually agreed between the interested parties.

5.2 The test specimens shall be carefully cleaned prior to testing so as to remove at best those traces (dirt, oil or other foreign matter) which could influence the result. The cleaning method used shall depend on the nature of the material, its surface and the contaminants, and shall not include the use of any abrasives or solvents, which may attack the surface of the specimens.

Thoroughly clean the specimens with an appropriate organic solvent (hydrocarbon, with a boiling point between 60 °C and 120 °C) using a clean soft brush or an ultrasonic cleaning device. Carry out the cleaning in a vessel full of solvent. After cleaning, rinse the specimens with fresh solvent, then dry them.

Care shall be taken that specimens are not recontaminated by careless handling after cleaning.

Specimens intentionally coated with protective organic films should not be cleaned prior to the test.

5.3 If the test specimens are cut from a larger coated article, cutting shall be carried out in such a way that the coating is not damaged in the area adjacent to the cut. Unless otherwise specified, the cut edges shall be adequately protected by coating them with a suitable material, stable under the conditions of the test, such as paint, wax or adhesive tape.

6 Arrangement of the test specimens

6.1 The test specimens shall be placed in the cabinet so that they are not in the direct line of travel of spray from the atomizer.

6.2 The angle at which the surface of the test specimens are exposed in the cabinet is very important. The specimen shall, in principle, be flat and placed in the cabinet facing upwards at an angle as close as possible to 20° to the vertical. This angle shall, in all cases, be within limits 15° to 25°. In the case of irregular surfaces, e.g. entire components, these limits shall be adhered to as closely as possible.

6.3 The test specimens shall be arranged so that they do not come into contact with the cabinet and so that the surface to be tested is exposed to free circulation of spray. The specimens may be placed at different levels within the cabinet, as long as the solution does not drip from specimens or their supports at one level on to other specimens placed below. However, for a new examination, or for tests with a total duration exceeding 96 h, location permutation of specimens is permitted. In this case, the number and frequency of the permutations are left to the operator and are to be indicated in the test report.

6.4 The supports for the test specimens shall be made of inert non-metallic material, such as glass, plastic or suitably coated wood. If it is necessary to suspend specimens, the material used shall on no account be metallic but shall be synthetic fibre, cotton thread or other inert insulating material.

7 Operating conditions

7.1 The test conditions are summarized in Table 1

Table 1 — Test conditions

1	Salt mist conditions (1) Temperature (2) Salt solution	35 °C ± 2 °C 50 g/l ± 5 g/l NaCl solution
2	“Dry” conditions (The air is purged under dry conditions) (1) Temperature (2) Relative humidity	60 °C ± 2 °C < 30 % RH
3	“Wet” conditions (Condensation on the test specimens shall not occur under wet conditions) (1) Temperature (2) Relative Humidity	50 °C ± 2 °C > 95 % RH
4	Period and content of a single exposure cycle	Total period 8 h, as follows: Salt mist spray 2 h “Dry” conditions 4 h “Wet” conditions 2 h (These times include the time for reaching the specified temperature for each condition)
5	Time to reach the specified condition (i.e. period taken for temperature and humidity to reach the specified values once the test condition has begun)	Mist to “Dry” < 30 min “Dry” to “Wet” < 15 min “Wet” to Mist < 30 min (Mist conditions are attained almost instantaneously once this condition begins)
6	Angle at which test specimens are supported	20° to the vertical

7.2 The test shall not be started until it has been confirmed, with the cabinet filled with dummy test specimens, that when the apparatus is operated in the salt mist condition the collection rate of solution measured over a period of 24 h of continuous spraying is within the range 1 ml/h to 2 ml/h for a horizontal collecting area of 80 cm² and that other conditions are as specified. In particular, the solution collected in each of the collecting devices shall have a sodium chloride concentration of 50 g/l \pm 5 g/l and a pH value in the range 6,5 to 7,2.

7.3 Test solution that has been sprayed shall not be re-used.

7.4 During the salt mist conditions of the test, the change from the set pressure shall be within \pm 0,002 5 MPa.

7.5 To check the reproducibility of test results, it is necessary to verify the corrosivity of test at regular intervals. A suitable method for evaluation of the corrosivity of test by use of reference specimens is described in annex B.

8 Continuity of tests

Tests shall preferably be continued without interruption throughout the test duration. When it is necessary to interrupt operation for the removal of specimens for inspection, the period of interruption should be minimized.

Where it is necessary to interrupt tests for a longer period, the test specimens shall be taken out from the test cabinet and treated the same way as after test (see clause 10), and thereafter stored in a desiccator until testing is resumed.

9 Duration of tests

The period of test shall be as designated by the specification covering the material or product being tested. When not specified, the period shall be agreed by the interested parties.

Recommended periods of exposure are 30 cycles (240 h), 45 cycles (360 h), 60 cycles (480 h), 90 cycles (720 h), 180 cycles (1 440 h).

10 Treatment of specimen after testing

At the end of the test period, remove the test specimens from the cabinet and allow them to dry in the open air for 0,5 h to 1 h before rinsing, in order to reduce the risk of removing corrosion products. Before they are examined, carefully remove the residues of spray solution from their surfaces. This may be accomplished by dipping the test specimens gently in clean running water at a temperature not exceeding 40 °C and then drying them immediately in a stream of air at a pressure not exceeding 200 kPa and at a distance of approximately 300 mm.

11 Evaluation of results

Many different criteria for the evaluation of the test results may be applied to meet particular requirements, e.g.:

- a) appearance after the test;
- b) appearance after removing superficial corrosion products;
- c) number and distribution of corrosion defects, i.e. pits, cracks, blisters, etc.; these may be assessed by methods described in ISO 10289;
- d) the time elapsing before the appearance of the first signs of corrosion;
- e) change in mass (see ISO 8407);
- f) alteration revealed by micrographic examination;
- g) change in mechanical properties;

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

12 Test report

The test report shall contain the following information:

- a) reference to this International Standard, i.e. ISO 14993;
- b) description of the test equipment;
- c) description of the material tested;
- d) dimensions and shape of the test specimen and nature and area of the surface tested;
- e) preparation of the test specimen, including any cleaning treatment applied before testing and any protection given to edges;
- f) temperature and relative humidity in each of the salt mist, "dry" and "wet" conditions during testing;
- g) the time taken to undergo the transitions from mist to "dry", "dry" to "wet", and "wet" to salt mist conditions during the test;
- h) mist collection rate, salt concentration and pH of the solution collected during the pre-test;
- i) the corrosivity of test determined as e.g. described in annex B;
- j) the frequency and duration of any interruptions;
- k) the number of cycles or the duration of the test;
- l) the method used to clean the test specimen after testing and any loss resulting from the cleaning operation and the method used for calibrating this mass loss;
- m) test results such as mass and thickness losses for uncoated specimens and blister width and peeling width for coated specimens;
- n) photographic records and/or descriptions of the appearance of tested specimens.

Annex A
(informative)

Typical apparatus for cyclic salt mist, “dry” and “wet” corrosion tests

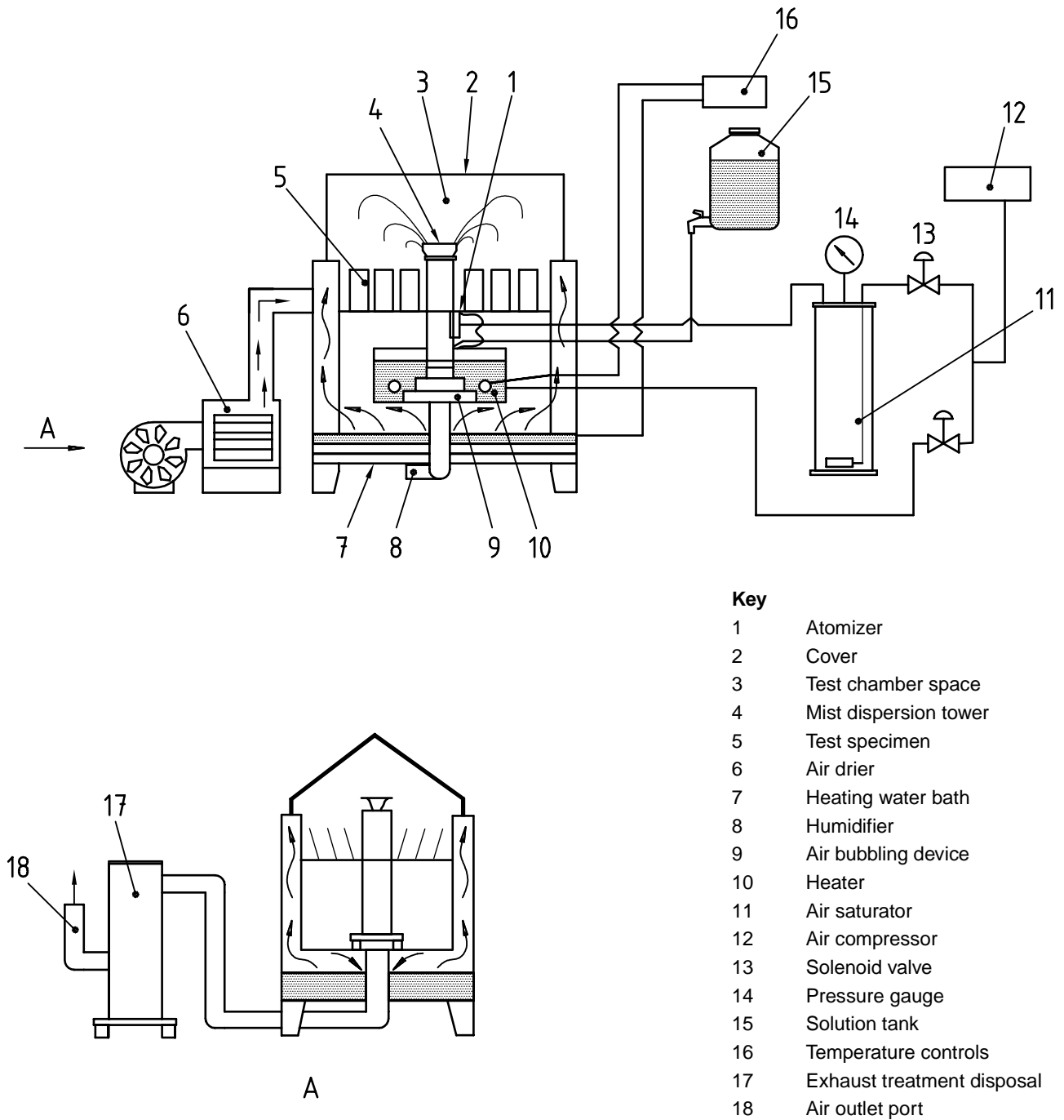


Figure A.1

Annex B (informative)

Method for evaluation of corrosivity of test

B.1 Reference test specimen

For measurement of the corrosivity in the climate cabinet during the test in accordance with this International Standard, use four reference specimens each of

- a) CR 4 grade steel according to ISO 3574, with a surface free from pores, slights, marks and scratches, and with a mat finish (arithmetical mean deviation of the profile $Ra = 0,8 \mu\text{m} \pm 0,3 \mu\text{m}$) and
- b) zinc with an impurity level of less than a mass fraction of 0,1 %.

The reference specimens should have a dimension of 150 mm × 70 mm × 1 mm. Before testing, the reference specimens shall be carefully cleaned with a hydrocarbon solvent in order to remove all marks of dirt, oil or other foreign matter capable of influencing the result of the corrosion rate determination. After drying, it shall be possible to weigh the reference specimens with a precision of at least 0,1 mg.

Protect one face of the reference specimens with a removable coating, e.g. an adhesive plastic film.

B.2 Arrangement of the reference specimens

Position the four reference specimens of each material in four different quadrants in the climate cabinet, with the unprotected face upward and at an angle of $20^\circ \pm 5^\circ$ from the vertical.

The support for the reference specimens shall be made of, or coated with, inert materials such as plastic and be placed at the same level as the test specimens.

B.3 Test duration

For the evaluation of the corrosivity of the test a test duration of 48 h should be used.

B.4 Determination of mass loss

Immediately after the end of the test, firstly remove the protective coating. Then remove the corrosion products by repetitive cleaning as described in ISO 8407. Use the following chemical cleaning procedures.

- a) For carbon steel a solution obtained by adding to hydrochloric acid ($\rho_{20} = 1,18 \text{ g/ml}$), 20 g of antimony trioxide (Sb_2O_3) per 1 000 g of hydrochloric acid and 50 g of tin(II) chloride (SnCl_2) per 1 000 g of hydrochloric acid.
- b) For zinc a solution of 5 % by mass of acetic acid in distilled water.

The chemical cleaning procedure is in both cases preferably performed in repetitive steps of immersion of 2 min. After each immersion step, thoroughly clean the reference specimen at ambient temperature under rinsing water with light brushing, then with acetone or ethanol, followed by drying. Weigh the reference specimen to the nearest 1 mg and plot the mass versus the actual cleaning cycle as described in ISO 8407.

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

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

mass loss per square metre into corrosion depth in μm by dividing by the density of the metal (density of carbon steel = $7,86 \text{ g/cm}^3$ and density of zinc = $7,14 \text{ g/cm}^3$).

B.5 Satisfactory operation of test apparatus

The test apparatus shall be considered to operate satisfactorily if after 48 h of testing, the loss in mass of each specimen is within the range:

- a) $200 \text{ g/m}^2 \pm 40 \text{ g/m}^2$ for the reference specimens of carbon steel;
- b) $40 \text{ g/m}^2 \pm 10 \text{ g/m}^2$ for the reference specimens of zinc.

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