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Corrosion of metals and alloys — Sulfur dioxide test in a humid atmosphere (fixed gas method)

Corrosion des métaux et alliages — Essai au dioxyde de soufre en atmosphère humide (méthode avec volume fixe de gaz)

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Foreword

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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 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*.

This first edition of ISO 22479 cancels and replaces ISO 3231:1993 and ISO 6988:1985, which have been combined and technically revised. The main changes compared with the previous edition are as follows:

- the method of generating sulfur dioxide from reagents has been deleted because of the risk of exposure to toxic chemicals.

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

Introduction

A humid atmosphere containing sulfur dioxide induces corrosion of many metals.

The results obtained in this document should not be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Similarly, performances of different materials in this document should not be taken as a direct guide to the relative corrosion resistance of these materials in service.

It is appropriate to test only the same corrosion protection systems at the same time in one test procedure, because an interaction between samples can't be prevented. When testing different corrosion protection systems with different materials, it should be taken into account that the influence of sulfur dioxide often can be different.

The term “fixed gas method” means that at the beginning of the test a fixed volume of gas is introduced into a cabinet of fixed volume.

Corrosion of metals and alloys — Sulfur dioxide test in a humid atmosphere (fixed gas method)

WARNING — This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices.

1 Scope

This document specifies a method for assessing the resistance of materials or products to a humid atmosphere containing sulfur dioxide.

This method is applicable to testing metals and alloys, metallic and non-organic coatings and organic coatings.

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 1514, *Paints and varnishes — Standard panels for testing*

ISO 2808, *Paints and varnishes — Determination of film thickness*

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:

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

4 Principle

The test specimens are exposed to a humid atmosphere containing sulfur dioxide. The sulfur dioxide dissolved by the moisture condenses on the test specimen surface and causes corrosion.

5 Apparatus

5.1 Component protection.

All components in contact with sulfur dioxide in a humid atmosphere shall be made of corrosion resistant materials, and shall themselves not emit any gas or vapour likely to influence corrosion of the test specimens.

5.2 Test cabinet.

The preferred capacity of the test cabinet is (300 ± 10) l. When agreed between the interested parties, other capacities may be used. In these cases, the size and/or number of test specimens, the volume of gas and the quantity of water shall be properly arranged in accordance with the capacity of the test cabinet.

The upper part of the cabinet shall be designed so that drops of condensed water formed on its surface do not fall on the test specimens being tested. An inclination of the upper part of the test cabinet of about 12° or more to the horizontal plane provides a suitable safeguard. The test specimens may be placed at different levels within the cabinet, as long as the solution does not drip from the test specimens or their supports at one level onto other test specimens placed below.

The temperature in the test cabinet is controlled by heating the floor and lower part of the side walls of the test cabinet. The temperature shall be measured at least 250 mm from the side walls and at least 150 mm below the upper part (lid).

A gas inlet port shall be less than 50 mm above the water surface. The test cabinet shall be hermetically leak-tight.

A pressure relief valve shall be placed in or near the upper part of the test cabinet.

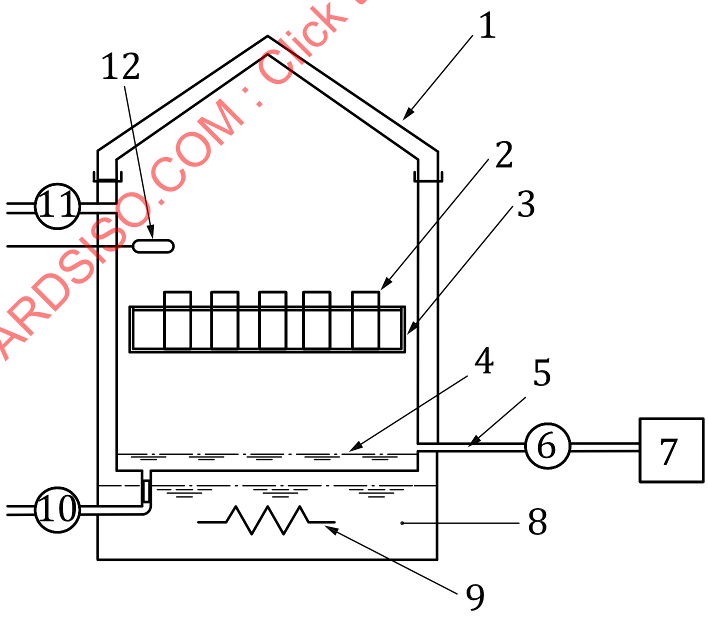
The exhaust gas and the released gas from the pressure relief valve shall be treated as appropriate.

NOTE Relevant regulatory limitations can apply.

A drain port shall be provided in the test cabinet.

A typical test cabinet is shown in Figure 1. Another example of test cabinet is shown in Figure A.1.

The test cabinet shall be installed in a room free from dust, draughts, corrosive gas and direct solar radiation, at a room temperature of (23 ± 5) °C and at a relative humidity of less than 75 %.



Key

1	lid	5	gas inlet port	9	heater
2	test specimens	6	flowmeter	10	drain port
3	specimen supports	7	source of sulfur dioxide	11	pressure relief valve
4	water inside the cabinet	8	water tank	12	temperature measurement

Figure 1 — Typical test cabinet

5.3 Source of sulfur dioxide.

The source of sulfur dioxide should be a gas cylinder with a volume concentration of more than 99,9 %. It shall be fitted with appropriate regulating and measuring apparatus to ensure the supply of the correct volume of gas. The volume of gas delivered into the test cabinet should be measured by a calibrated flow meter. Another measuring instrument works with squeezing out a viscous liquid paraffin for the volume of the gas for 0,2, 1,0 and 2,0 l.

WARNING — Sulfur dioxide (CAS no. 7446-09-5) is toxic, corrosive and irritating. Handling of sulfur dioxide shall be restricted to skilled personnel or conducted under their control. The apparatus 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.

5.4 Conditioning of a new cabinet.

In order to minimize the effect from the material of the cabinet by sulfur dioxide, a new cabinet shall be operated at least one test cycle in accordance with the procedures given in 8.2 to 8.5 without test specimens. The conditioning shall conform to Table 1, with 2,0 l of sulfur dioxide.

6 Test specimens

6.1 General

The number, type, surface roughness, thickness of coatings, shape and dimensions of test specimens shall be selected according to the specification for the materials or product being tested. When not specified, details concerning the test specimens shall be agreed between the interested parties.

6.2 Dimensions

A typical specimen size is 150 mm × 100 mm by 0,75 mm to 1,25 mm thickness.

6.3 Preparation

6.3.1 Metals and alloys

Thoroughly clean the test specimens before testing. The cleaning method depends on the nature of the surface and the contaminants. Abrasives shall not include the use of any abrasives or solvents that may attack the surface of the test specimens. Take care that the test specimens are not recontaminated after cleaning, by excessive or careless handling.

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

6.3.2 Paints and varnishes

Unless otherwise specified or agreed, the test specimens shall be prepared in accordance with ISO 1514 and then coated by the specified method with the product or system under test. The back and edges of the test specimens should be coated with the product or system under test.

Dry (or cure) and age (if applicable) each coated test specimen for the specified time and under the specified conditions and, unless otherwise specified, condition them at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) % for at least 16 h, with free circulation of air and not exposed to direct sunlight. The test procedure shall then be carried out as soon as possible.

Determine the thickness, in micrometres, of the dried coating by one of the non-destructive procedures described in ISO 2808.

6.4 Arrangement of the test specimens

The distance between test specimens shall be not less than 20 mm. The distance between the edge of the test specimens and a wall of the test cabinet shall be not less than 100 mm. The test specimens shall be arranged in such a way that they cannot protect each other from the influence of the environment. The distance between the lowest edge of the test specimens and the surface of the water shall be greater than 200 mm. The contact area between test specimens and their supports shall be as small as possible.

The orientation of the exposed test surface in the test cabinet is critical. Unless otherwise specified, test specimen shall be mounted at an angle between vertical and (15 ± 2)° to the vertical, with the area of primary interest facing up.

NOTE This is common practice for testing metallic specimens and measures to protect them from corrosion. When testing assemblies or enclosures, it is preferable to mount the specimen at the same angle as in typical use.

The total exposed surface area of the test specimens for one test shall be (0,5 ± 0,1) m^2 for each (300 ± 10) l of the test cabinet. For different volumes of the test cabinet, the total exposed surface area is to be adjusted proportionally. When the total exposed surface area is less than 0,5 m^2 for 300 l of the cabinet, substitute specimens shall be added to obtain a total surface area of 0,5 m^2. The materials of the substitute specimens shall be the same as of the test specimens. Otherwise, test results may be different, since the influence (absorbance) of sulfur dioxide differs by materials.

7 Test conditions

7.1 Test cycles

One test cycle is 24 h, either Method A or Method B.

In Method A, test specimens are exposed to sulfur dioxide for 24 h. If test specimens are affected seriously by the temperature and humidity, Method B should be chosen.

In Method B, test specimens are exposed to sulfur dioxide for 8 h, followed by exposure to a standard atmosphere for 16 h.

The test conditions of Method A and Method B are shown in Table 1 and Table 2, respectively.

For metallic and non-organic coatings, 0,2 l volume of sulfur dioxide should be used. For paint and varnish, 0,2 l or 1,0 l volume of sulfur dioxide should be used. Generally, 0,2 l volume of sulfur dioxide is recommended where the coating thickness does not exceed approximately 40 µm. 2,0 l volume of sulfur dioxide is used by agreement between the interested parties or according to product specifications, such as corrosion resistant coatings (e.g. hard chrome plating, anodised coating).

Table 1 — Test conditions of Method A

Conditions	Sulfur dioxide
One test cycle	24 h
Temperature	(40 ± 3) °C
Relative humidity	Approximately 100 %
Volume of sulfur dioxide	0,2 l, 1,0 l or 2,0 l [at (300 ± 10) l of capacity]
Quantity of water	(2,0 ± 0,2) l [at (300 ± 10) l of capacity]

NOTE 1 Theoretical concentrations of sulfur dioxide at the beginning of each test cycle are 0,067 %, 0,33 % and 0,67 %, corresponding to 0,2 l, 1,0 l and 2,0 l of sulfur dioxide gas, respectively. However, much of the sulfur dioxide is quickly dissolved into the water at the bottom of the test cabinet. Thus, the effective real sulfur dioxide concentration in the gas cabinet is much lower than the theoretical concentration (see Figure C.1).

NOTE 2 The ± tolerances given for the temperature are allowable fluctuations, which are defined as the positive and negative deviation from the setting of the sensor at the operational control set point during equilibrium conditions. This does not mean that the set value can vary by plus/minus the amount indicated from the given value.

Table 2 — Test conditions of Method B

Conditions	Sulfur dioxide	Standard atmosphere
Time of exposure	8 h	16 h
One test cycle	24 h	
Temperature	(40 ± 3) °C	(23 ± 5) °C
Relative humidity	Approximately 100 %	Less than 75 %
Volume of sulfur dioxide	0,2 l, 1,0 l or 2,0 l [at (300 ± 10) l of capacity]	—
Quantity of water	(2,0 ± 0,2) l [at (300 ± 10) l of capacity]	

NOTE 1 Theoretical concentrations of sulfur dioxide at the beginning of each test cycle are 0,067 %, 0,33 % and 0,67 %, corresponding to 0,2 l, 1,0 l and 2,0 l of sulfur dioxide gas, respectively. However, much of the sulfur dioxide is quickly dissolved into the water at the bottom of the test cabinet. Thus, the effective real sulfur dioxide concentration in the gas cabinet is much lower than the theoretical concentration (see Figure C.1).

NOTE 2 The ± tolerances given for the temperature are allowable fluctuations, which are defined as the positive and negative deviation from the setting of the sensor at the operational control set point during equilibrium conditions. This does not mean that the set value can vary by plus/minus the amount indicated from the given value.

7.2 Test duration

The test duration shall be as designated by the specification for the material or product being tested. When not specified, the test duration shall be agreed between the interested parties.

The test may be finished if a specified degree of corrosion has been reached, or if the appearance or function of test specimens has otherwise been impaired to an unacceptable level.

Recommended numbers of test cycles are: 1, 2, 5, 10, 15 or 20.

8 Procedure

8.1 Introduction of water

Introduce (2,0 ± 0,2) l of distilled or deionized water with a conductivity not higher than 5 µS/cm at (25 ± 2) °C inside the test cabinet.

NOTE This quantity is for 300 l capacity of the test cabinet. It depends on the capacity of the test cabinet proportionally (see 5.2).

8.2 Arrangement of the test specimens

Place the test specimens in position (see 6.4), and close tightly the test cabinet.

8.3 Introduction of sulfur dioxide

Introduce 0,2 l, 1,0 l or 2,0 l of sulfur dioxide at (23 ± 5) °C into the test cabinet through the gas inlet port.

NOTE This volume is for 300 l capacity of the test cabinet. It depends on the capacity of the test cabinet proportionally (see 5.2).

The test cycle begins when sulfur dioxide is introduced.

8.4 Heating the test cabinet

The temperature of the test cabinet shall be raised to (40 ± 3) °C in less than 1,5 h and maintained for the specified period.

8.5 Introduction of standard atmosphere

In the case of Method B, the standard atmosphere is introduced by turning off the heater while opening and/or ventilating the test cabinet.

The temperature of the test cabinet shall be lowered to (23 ± 5) °C, less than 75 % relative humidity in approximately 1 h and be maintained for the specified period.

Alternatively, the test specimens may be transferred to a different environmental cabinet capable of being maintained at the conditions of standard atmosphere.

8.6 Replacement of water and sulfur dioxide

The water and the sulfur dioxide in the test cabinet shall be replaced for each test cycle (24 h).

8.7 Cleaning of test specimens after test

8.7.1 General

Procedures for treatment of test specimens after testing shall be agreed between the interested parties before starting the test.

8.7.2 Metals and alloys, metallic and non-organic coating test specimens

At the end of the test cycle, remove the test specimens from the cabinet. Before they are examined, allow them to hang freely in a normal indoor atmosphere until any liquid corrosion products have solidified. First, examine them with all corrosion products in position. Any cleaning carried out shall depend on the criteria laid down for the evaluation of the results of the test.

8.7.3 Organic coating test specimens

At the end of the test cycle, remove the test specimens from the cabinet, blot them with absorbent paper and immediately examine the whole test surface of each test specimen for corrosion defects. Allow the test specimens to stand in the open at room temperature for 24 h. Then examine the test surface. If it is required to examine the substrate for signs of attack, remove the coating according to the specification for the materials or product being tested.

8.8 Performance check

In order to ensure the reproducibility and repeatability of test results obtained either with one or multiple test cabinets, performance checks shall be carried out. A suitable method for evaluation of the performance of the test cabinet by use of reference specimens is described in Annex B.

9 Evaluation

Several different criteria for evaluation of the test results may be used to meet particular requirements, for example:

a) appearance after test as described in the ISO 4628 series and ISO 10289;

b) appearance after removing superficial corrosion products described in the ISO 4628 series and ISO 10289;

c) number and distribution or corrosion defects, i.e. pits, cracks, blisters:

    1) in the case of metals and alloys, metallic and non-organic coatings be assessed by methods described in ISO 10289;

    2) in the case of organic coatings, those be assessed by methods described in the ISO 4628 series;

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

e) change in mass;

f) alteration revealed by micrographic examination;

g) change in mechanical properties.

10 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 22479:2019;

b) the concentration of sulfur dioxide used;

c) whether the test was performed in accordance with Method A or Method B (see 7.1);

d) the specification of the base material;

e) the type, dimensions, description and (if necessary) surface roughness of the test specimens;

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

g) the type of coating with an indication of its surface finish;

h) the item of information related to the test specimen of paint and varnishes:

    1) duration and conditions of drying (or curing) and ageing (if applicable) the coating before test;

    2) thickness of the dry coating and method of measurement;

i) the number of test specimens of each coating or product subjected to the test;

j) the methods, if any, used to clean the test specimens after test, with, when appropriate, an indication of the loss of mass resulting from the cleaning operation;

k) the corrosivity of test determined as, for example, described in Annex B;

l) the temperature reading within the exposure zone of the test cabinet;

m) the test duration;

n) the angle of inclination of the test specimens during exposure;

o) the character of any test specimens placed in the cabinet expressly to check the correctness of the operating conditions and the results obtained;

p) the results of all inspections;

q) any deviations from the procedure;

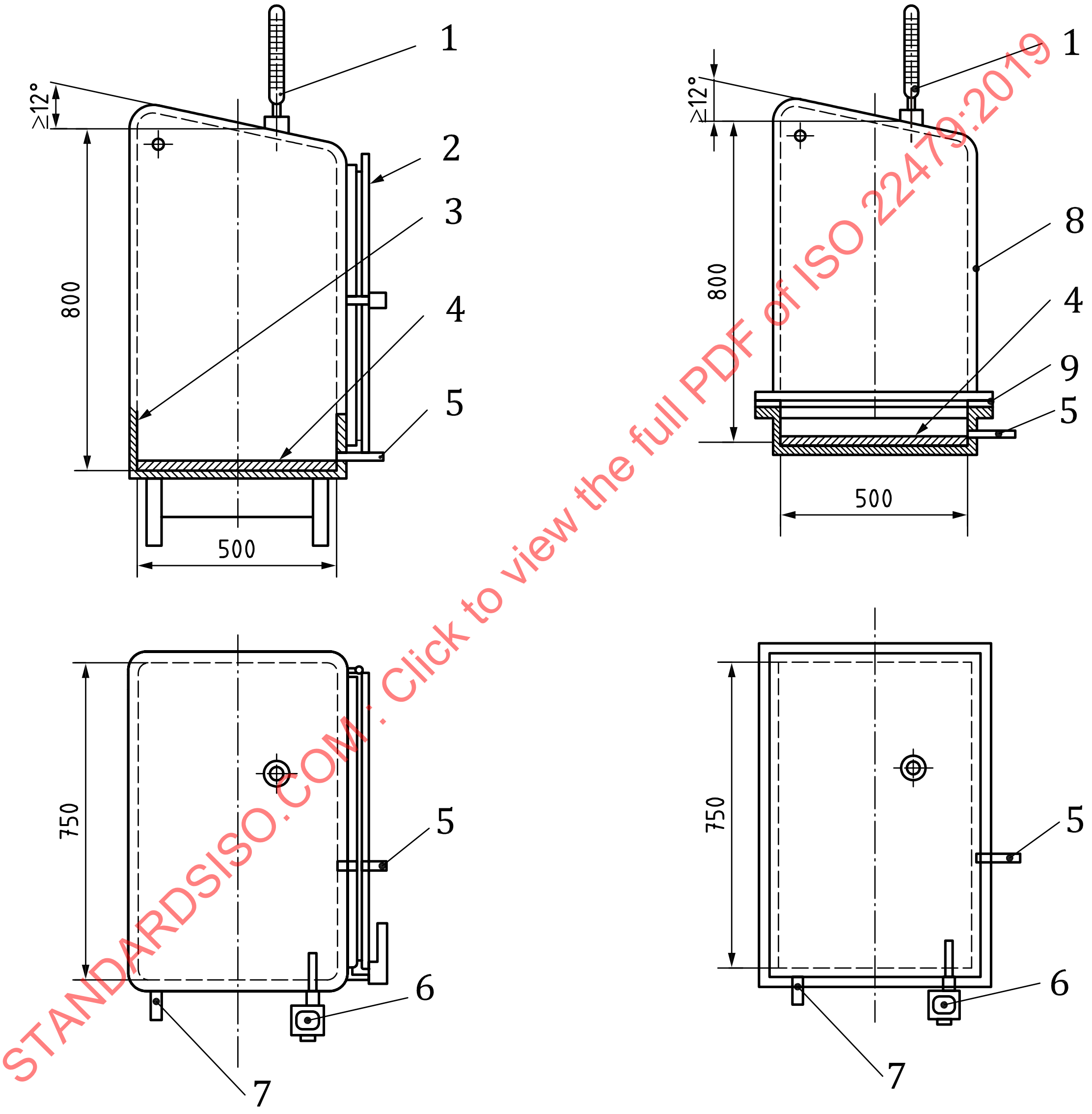
r) any unusual features observed;

s) the date of the test.

Annex A
(informative)

Another example of a test cabinet

Dimensions in millimetres



a) Cabinet with door **b) Cabinet with hood**

Key

1	thermometer
2	door
3	water trough
4	water surface
5	gas inlet
6	temperature regulating device
7	pressure relief valve
8	hood
9	sealing strip

Figure A.1 — Another example of a test cabinet

Annex B
(informative)

Performance check

B.1 General

In order to ensure the reproducibility and repeatability of test results obtained either with one or several test apparatuses, performance checks may be performed.

This annex defines a test cabinet with 300 l capacity with 0,2 l volume of sulfur dioxide.

B.2 Reference specimens

The performance check is carried out using five reference specimens made of CR4-grade steel in accordance with ISO 3574. The dimensions of the reference specimens are 50 mm × 100 mm by 0,6 mm to 1,5 mm thickness.

In order to meet the requirement of total exposed surface area, (0,5 ± 0,1) m^2 (see 6.4), two substitute specimens made of CR4-grade steel in accordance with ISO 3574 are required. The dimensions are 250 mm × 400 mm × 1 mm.

B.3 Preparation of reference specimens

Immediately prior to the test, clean the reference and substitute specimens carefully by eliminating all those traces (dirt, oil or other foreign matter) influencing the test results. Degrease with white spirit or some other suitable solvent using a soft, lint-free cloth or brush. Weigh the five reference specimens to an accuracy of 1 mg. If weighing cannot be made immediately after degreasing, they should be kept in a desiccator until weighing can be performed.

B.4 Procedure

B.4.1 Arrangement of reference specimens

The five reference specimens are placed vertically in the test cabinet and the two substitute specimens positioned vertically on either side of them in accordance with 6.4.

B.4.2 Test conditions

The five test cycles shall be performed in accordance with test conditions of Method B in Table 2, with 0,2 l of sulfur dioxide.

B.4.3 Removal of corrosion products

At the end of the test, immediately take the reference specimens out of the test cabinet. The corrosion products can be removed from the reference specimens by pickling carried out at a temperature of 18 °C to 28 °C. Then, hydrochloric acid with a density ρ = 1,10 g/ml and inhibited with 3,5 g hexamethylenetetramine per litre (e.g. by making up 500 ml of chemically pure HCI, ρ = 1,19 g/ml, to 1 l with distilled or deionized water) or 200 g of diammonium hydrogen citrate $[(NH_4)_2HC_6H_5O_7]$ (recognized analytical grade) per litre in distilled or deionized water for 10 min at 23 °C can be used.

Other solutions as specified in ISO 8407 may be used.

B.4.4 Weighing of reference specimens

After removal of the corrosion products, the reference specimens are rinsed thoroughly in distilled or deionized water and dried. They are weighed to an accuracy of 1 mg. If they cannot be weighed immediately after drying, they are to be kept in a desiccator until weighing can be performed.

B.5 Expression of results

The loss in mass determined shall be expressed as a function of the total area exposed to corrosion, in g/m^2. For a successful performance check, the mean value shall be (145 ± 35) g/m^2, with individual values not deviating from the mean value by more than 20 %.