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Corrosion of metals and alloys — Accelerated cyclic tests with exposure to acidified salt spray, “dry” and “wet” conditions

Corrosion des métaux et alliages — Essais cycliques accélérés avec exposition au brouillard salin acidifié, en conditions «sèches» et en conditions «humides»

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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 16151 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

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

The accelerated corrosion-test methods described in this International Standard are designed to simulate and enhance the environmental influence on a metallic material to outdoor climates, where exposure to acid rain and to salt-contaminated conditions occur and may promote corrosion. It has been prepared by reference to technical papers and reports (see the Bibliography).

The test methods involve cyclic exposure of test specimens to a mist of acidified-salt solution, to drying conditions, and to periods of high humidity. However, the methods are 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 in which they may be used. Nevertheless, the methods provide valuable information on the relative performance of materials exposed to salt/acid rain environments similar to those employed in the test.

Corrosion of metals and alloys — Accelerated cyclic tests with exposure to acidified salt spray, “dry” and “wet” conditions

1 Scope

This International Standard specifies two accelerated corrosion-test procedures, Methods A and B, for the comparative evaluation of metallic materials with or without permanent corrosion protection or temporary corrosion protection in outdoor salt/acid rain environments. It also specifies the apparatus used. The two tests involve cyclic exposure of the specimens to acidified salt spray, “dry” and “wet” conditions.

The particular advantages of the two tests over conventional accelerated tests, such as the neutral salt spray test (NSS) as specified in ISO 9227 lie in their better ability to reproduce the corrosion that occurs in outdoor salt/acid rain environments. They are also useful for evaluating cosmetic corrosion.

Method A applies to

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

Method B applies to

- steel coated with anodic coatings, and
- steel coated with anodic coatings covered with conversion coatings.

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 4628-1:2003, *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 4628-2:2003, *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 4628-3:2003, *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 4628-4:2003, *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 4628-5:2003, *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 8407:1991[1], *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

ISO 8993:1989, *Anodized aluminium and aluminium alloys — Rating system for the evaluation of pitting corrosion — Chart method*

ISO 9227:1990[1], *Corrosion tests in artificial atmospheres — Salt spray tests*

ISO 10289:1999, *Method for corrosion testing of metallic and other inorganic coatings on metallic substrate — Rating of test specimens and manufactured articles subjected to corrosion tests*

ISO 11130:1999, *Corrosion of metals and alloys — Alternate immersion test in salt solution*

3 Test solution

The following clauses give instructions for the preparation and use of the solutions used in Methods A and B.

3.1 Method A

3.1.1 Preparation of acidic 5 % sodium chloride solution

3.1.1.1 Neutral 5 % sodium chloride solution

A sufficient mass of sodium chloride shall be dissolved in distilled or deionized water, with a conductivity not higher than 20 µS/cm at 25 °C ± 2 °C, to produce a concentration of 50 g/l ± 5 g/l. The specific gravity range for a 50 g/l ± 5 g/l solution shall be 1,029 to 1,036 at 25 °C.

The sodium chloride shall contain less than 0,001 % mass fraction of copper and less than 0,001 % 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 % mass fraction of sodium iodide, or more than a mass fraction of total impurities of 0,5 % calculated for dry salt.

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

3.1.1.2 Acidification

The pH of the solution shall be adjusted to a value of 3,5 ± 0,1 at 25 °C ± 2 °C, by adding the following reagents to 10 litres of the prepared neutral 5 % sodium chloride solution as follows:

— 12 ml of nitric acid (HNO_3, $\rho = 1,42$ g/ml);

— 17,3 ml of sulfuric acid (H_2SO_4, $\rho = 1,84$ g/ml);

— a sufficient quantity of 10 % mass fraction of sodium hydroxide (NaOH) solution, to adjust the pH of the solution to 3,5 ± 0,1 (about 300 ml will be required).

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[1] Under revision.

3.2 Method B

3.2.1 Preparation of the mixed salt solution

The mass of reagent shown in Table 1 shall be dissolved in distilled or deionized water, with a conductivity not higher than 20 µS/cm at 25 °C ± 2 °C, to produce a stock solution with a concentration of 36 g/l ± 3,6 g/l. It shall be diluted by 1:6 to produce a mixed salt solution with a concentration of 6,0 g/l ± 0,6 g/l.

The composition of the stock solution is the same as a typical synthetic ocean water shown in ISO 11130:1999, Annex A, A.3 Test solution for simulating the corrosive effects of ocean water.

Table 1 — Composition and concentration of a stock solution for a mixed salt solution

Reagents	Concentration g/l
NaCl	24,53
$MgCl_2$	5,20
Na_2SO_4	4,09
$CaCl_2$	1,16
KCl	0,695
$NaHCO_3$	0,201
KBr	0,101
H_3BO_3	0,027
$SrCl_2$	0,025
NaF	0,003
WARNING — Handling of $SrCl_2$ and NaF can be hazardous and shall be resticted to skilled chemists or conducted under their control.	

3.2.2 Preparation of the acidic solution

To prepare the acidic solution, 16,2 g of concentrated nitric acid (HNO_3, ρ = 1,40 g/ml, with a mass fraction of HNO_3 equal to 0,65) and 42,5 g of sulfuric acid (H_2SO_4, ρ = 1,84 g/ml, with a mass fraction of H_2SO_4 equal to 0,96) shall be dissolved in water and diluted to a total volume of 1 litre to make 1 N acid solution with respect to nitric acid and sulfuric acid at an equivalent ratio of $[NO_3^-]/[SO_4^{2-}]$ of 0,4.

3.2.3 Preparation of the acidified-salt solution

The prepared acidic solution from 3.2.2 shall be added to the mixed salt solution from 3.2.1 to adjust the pH to 2,5 ± 0,1 at 25 °C ± 2 °C.

NOTE The relationship between the amount of mixed acidic solution from 3.2.2 and the pH of the acidified-salt solution is shown in Annex A. This solution, with pH value near to 2,5, would have a no-buffering action.

4 Apparatus

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

4.1 Exposure cabinet

The exposure cabinet shall have a minimum capacity of 0,4 m^3. For large-capacity cabinets, during periods of exposure to acidified salt spray, it shall be ensured that the necessary condition 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 rate of collection 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 accelerated cyclic tests with exposure to acidified salt spray, “dry” and “wet” conditions is shown in Annex B.

4.2 Humidity and temperature control

An appropriate system shall be used to maintain 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.

4.3 Spraying device

The device for spraying the acidified-salt solution shall comprise a supply of clean air, of controlled pressure and humidity, 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 the atomizing pressure shall be at an overpressure of 70 kPa to 170 kPa, and preferably 98 kPa constant.

4.4 Air saturator

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

During periods of exposure to the acidified-salt solution 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 within the specified limits (see 7.2). The level of water shall be maintained automatically to ensure adequate humidification.

The atomizers shall be made of inert material, for example glass or plastic materials. Baffles may be used to prevent direct impact of spray on the test specimens and the use of a modern mist dispersion tower is preferable in obtaining constant distribution of spray within the cabinet. The level of the acidified-salt solution in the supply tank shall be maintained automatically to ensure constant spray delivery throughout the test.

4.5 Collecting devices

At least two suitable collecting devices shall be available, consisting of clean funnels with a collecting area of approximately 80 cm^2, 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, at least one close to the spray inlet and one remote from the inlet and in such a way that only spray, and not the liquid falling from specimens or from parts of the cabinet, is collected.

4.6 Air dryer

A heating device and fan shall supply dried air of the specified humidity during “dry” periods (see Tables 2 and 3).

4.7 Exhaust system

The system by which air is exhausted from the spray cabinet shall not be influenced by the back pressure of the atmosphere when the air is released at a point outside the building. It is preferred that the apparatus has a means to properly treat the fog prior to releasing it outside the building (see Annex B).

4.8 Drain system

An appropriate system to treat the drain water shall be provided, so that the solution is not directly discharged into the drainage system (see Annex B).

5 Test specimens

5.1 The number and type of test specimens 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 those traces (dirt, oil or other foreign matter), which could influence the result. The cleaning method employed shall depend on the nature of the material, its surface and the contaminants, but shall not include the use of any abrasives or solvents which may attack the surface of the specimens.

For metals or alloy without organic coatings, and inorganic coating materials, the specimens shall be thoroughly cleaned 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. The cleaning shall be carried out in a vessel full of solvent. After cleaning, the specimens shall be rinsed with fresh solvent, then dried.

Unless otherwise specified, specimens intentionally coated with protective organic films should not be cleaned prior to the test. If cleaning is necessary, specimens shall be wiped with gauze impregnated with ethanol, taking care not to damage the surface of the specimens.

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

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 If the cabinet is not full of test specimens, it is recommended to fill it with inert flat dummy specimens of the same size as a reference specimen, in order to ensure uniformity of the spray. The material used shall be plastics, glass, or other inert insulating materials, which would not influence corrosion of the test specimens being tested.

6.3 The angle at which the surfaces 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, for example entire components, these limits shall be adhered to as closely as possible.

6.4 The test specimens shall be arranged so that they do not come into contact with the cabinet and so that the surfaces to be tested are exposed to free downward falling 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 onto specimens placed below them. 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 shall be indicated in the test report.

6.5 The supports for the test specimens shall be made of inert non-metallic material, such as glass, plastics or other suitably coated wood. If it is necessary to suspend specimens, the material used shall not be metallic but shall be synthetic fibre, cotton thread or another inert insulating material.

7 Operating conditions

7.1 The test conditions used shall be in accordance with those given in Table 2 for Method A, and Table 3 for Method B.

Table 2 — Test conditions in Method A

1	**Acidified salt mist conditions**		
	1) Temperature	35 °C ± 1 °C	
	2) Acidified-salt solution	pH 3,5 ± 0,1, salt concentration 50 g/l ± 5 g/l as described in 3.1	
2	**“Dry” conditions**		
	1) Temperature	60 °C ± 1 °C	
	2) Relative humidity	< 30 % RH	
3	**“Wet” conditions**		
	1) Temperature	50 °C ± 1 °C	
	2) Relative humidity	> 95 % RH	
4	**Length and composition of a single exposure cycle**	Total period 8 h:	
		Acidified salt mist	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 conditions**	“Mist” to “Dry”	< 30 min
	(i.e. time taken for temperature and humidity to reach the specified values once the test conditions have been changed)	“Dry” to “Wet”	< 15 min
		“Wet” to “Mist”	< 30 min
		(“Mist” conditions shall be attained almost instantaneously after changing to these conditions)	
6	**Angle at which test specimens are supported**	20° ± 5° to the vertical	

Table 3 — Test condition in Method B

1	**Acidified salt mist conditions**		
	1) Temperature	35 °C ± 1 °C	
	2) Acidified-salt solution	pH 2,5 ± 0,1, salt concentration 6,0 g/l ± 0,6 g/l as described in 3.2	
2	**“Dry” conditions**		
	1) Temperature	60 °C ± 1 °C	
	2) Relative humidity	< 30 % RH	
3	**“Wet” conditions**		
	1) Temperature	40 °C ± 1 °C	
	2) Relative humidity	(85 ± 5) % RH	
4	**Length and composition of a single exposure cycle**	Total period 8 h:	
		Acidified salt mist	1 h
		“Dry” conditions	4 h
		“Wet” conditions	3 h
		(These times include the time for reaching the specified temperature for each condition)	
5	Time to reach the specified conditions	“Mist” to “Dry”	< 30 min
	(i.e. time taken for temperature and humidity to reach the specified values once the test conditions have been changed)	“Dry” to “Wet”	< 15 min
		“Wet” to “Mist”	< 30 min
		(“Mist” conditions shall be attained almost instantaneously after changing to these conditions)	
6	**Angle at which test specimens are supported**	20° ± 5° to the vertical	

7.2 In Method A, the test shall not be started until it has been confirmed, with the cabinet filled with inert flat dummy specimens, that, when the apparatus is operated in the acidified salt mist condition, the collection rate of solution measured over a period of 24 h of continuous spraying is within the range 1,5 ml/h ± 0,5 ml/h for a horizontal collecting area of 80 cm^2 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 ± 5 g/l and a pH in the range 3,4 to 3,6.

In Method B, the test shall not be started until it has been confirmed with the cabinet filled with inert flat dummy specimens, that, when the apparatus is continuously operated in the acidified salt mist condition, the collection rate of solution measured over a period of 24 h of continuous spraying is within the range 1,5 ml/h ± 0,2 ml/h for a horizontal collecting area of 80 cm^2 and that other conditions are as specified. In particular, the solution collected in the collecting devices shall have a salt concentration of 6,0 g/l ± 0,6 g/l and a pH in the range 2,4 to 2,6.

NOTE In Method B, as the test period for the acidified-salt solution mist in the accelerated cycle is only 1 h and the corrosion resistance of steels is so sensitive to salt concentration, it is necessary to control the collection of solution more strictly.

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

7.4 During the test, changes of cabinet pressure shall be avoided.

7.5 To check the reproducibility of the test results, the corrosivity of the test shall be verified at regular intervals. A suitable method for the evaluation of the corrosivity of the test chamber by use of reference specimens is described in Annex C.

8 Continuity of tests

Tests shall preferably be continued without interruption throughout the test duration. When it is necessary to interrupt the 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 treated as follows:

- In Method A, the test specimens shall be taken out from the test cabinet and treated in the manner described in Clause 10 of ISO 9227:1990, and thereafter stored in a desiccator until testing is resumed.
- In Method B, the test specimens shall be taken out from the test cabinet and dried as they are, and then stored in a desiccator until testing is resumed. When storing, care shall be taken not to remove or detach deposits and corrosion products on the surfaces of the test specimens. Accumulating salt will influence corrosion resistance and the specimens shall not be cleaned.

9 Duration of tests

9.1 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 test are:

a) For Method A: 3 cycles (24 h), 6 cycles (48 h), 12 cycles (96 h), 30 cycles (240 h), 45 cycles (360 h), 60 cycles (480 h), 90 cycles (720 h), 180 cycles (1 440 h).

b) For Method B: 12 cycles (96 h), 24 cycles (192 h), 36 cycles (288 h), 60 cycles (480 h), 96 cycles (768 h), 192 cycles (1 536 h).

10 Treatment of specimens after test

At the end of the test period, the test specimens shall be removed from the cabinet and allowed to dry for 0,5 h to 1 h before rinsing, in order to reduce the risk of removing corrosion products. Before they are examined, the residues of spray solution shall be carefully removed 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 an overpressure not exceeding 200 kPa and at a distance of approximately 300 mm.

NOTE If it is necessary to remove superficial corrosion products for evaluating changes in mass, this may be carried out by the methods described in ISO 8407.

Where superficial corrosion products on 55 % Al-Zn coatings are removed, it is recommended to apply the procedure for zinc specified in ISO 8407, and not for aluminium specified in the same International Standard.

11 Evaluation of results

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

a) appearance after the test;

b) appearance after removing superficial corrosion products;

c) number and distribution of corrosion defects (i.e. pits, cracks, blisters, rusting or creep from scratches in the case of organic coatings); these may be assessed by methods described in ISO 8993 or ISO 10289 and for organic coatings in ISO 4628, Parts 1 to 5);

d) the time elapsing before the appearance of the first signs of corrosion;

e) changes in mass;

f) alteration revealed by micrographic examination;

g) changes in mechanical or electrical 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 provide the following information:

a) reference to this International Standard;

b) description of the test equipment;

c) description of the material tested;

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

e) details of the 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 acidified-salt solution mist, “dry” and “wet” conditions during the test;

g) the time taken to undergo the transitions from “mist” condition to “dry”, “dry” to “wet”, and “wet” to “mist” conditions during the test;

h) mist collection rate, salt concentration, and pH of the solution collected during the pretest;

i) the frequency and duration of any interruptions;

j) the number of cycles or the duration of the test;

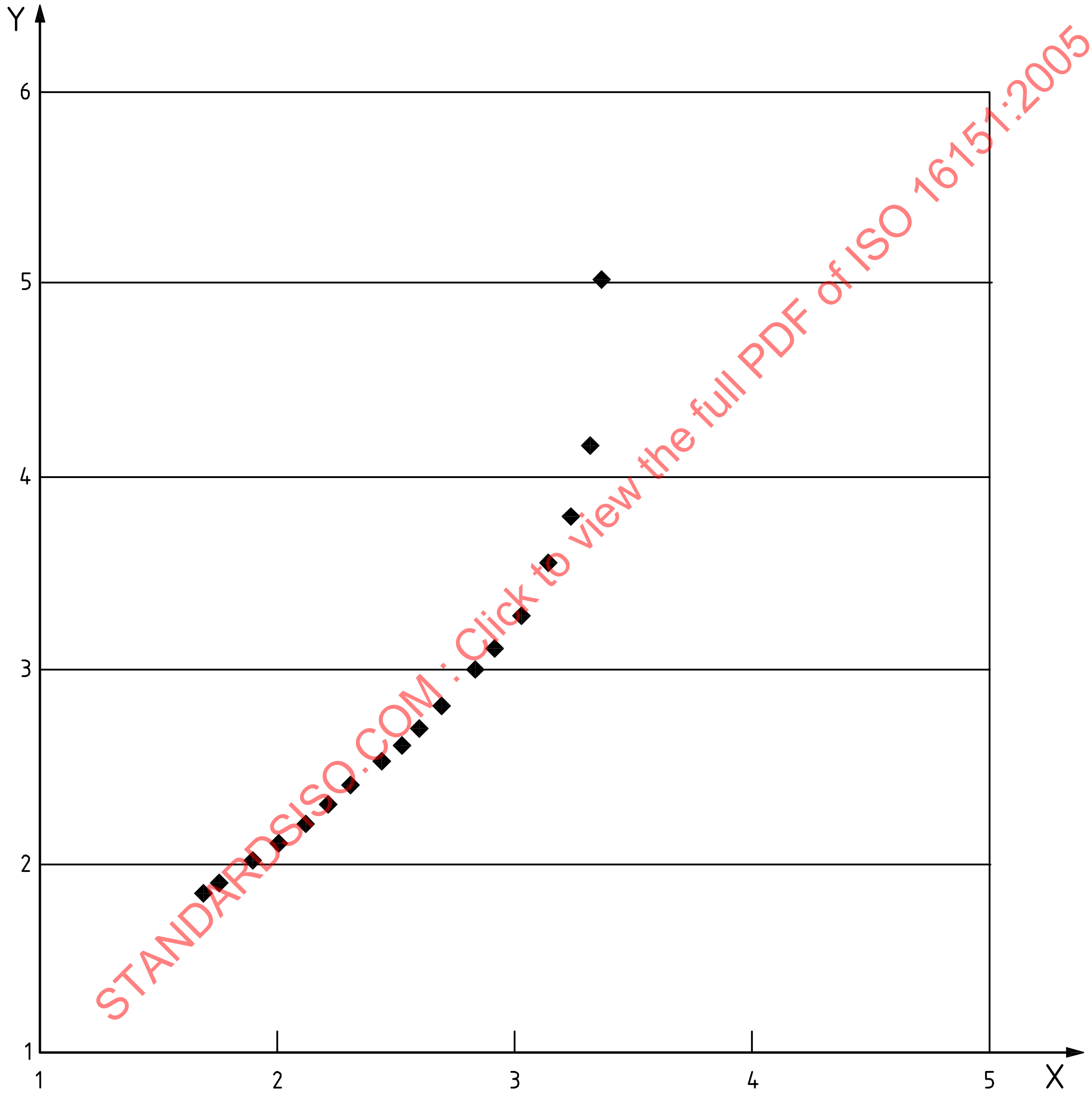
k) the method used to clean the test specimens after testing and, where appropriate, an indication of the mass loss resulting from the cleaning operation and the method used for correcting for this mass loss;

l) test results, such as mass and thickness losses for uncoated specimens and blister width and peeling width for coated specimens;

m) if necessary, photographic records and/or descriptions of the appearance of tested specimens.

Annex A
(informative)

Relationship between amount of acidic stock solution added to mixed salt solution and pH of the resulting acidified-salt solution



Key

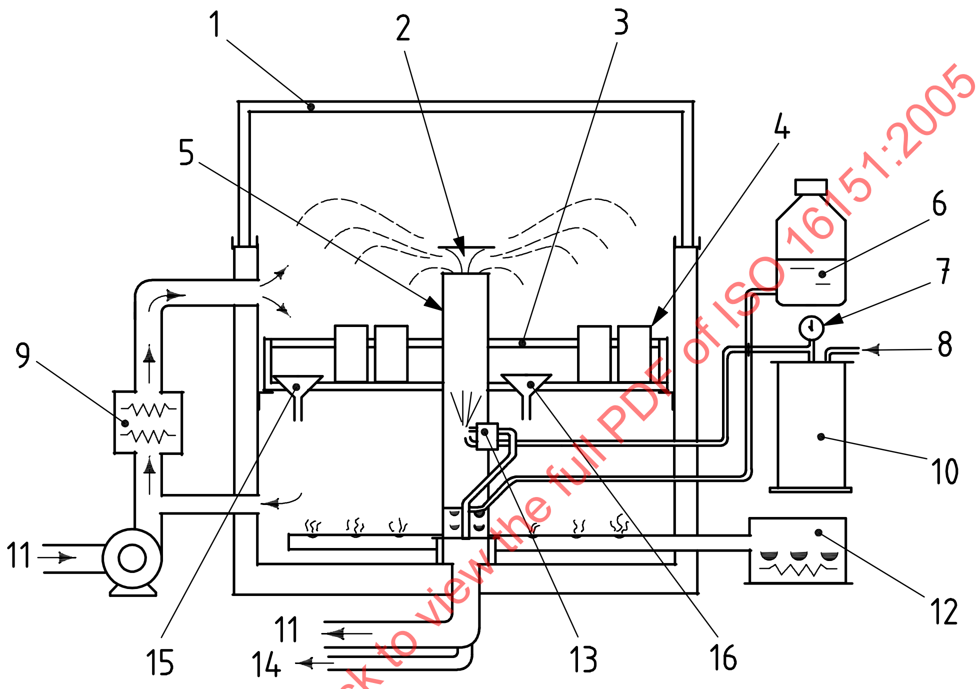
X amount of acidic stock solution added to mixed salt solution, expressed as – log [(added volume of the solution prepared in 3.2.2)/(volume of the solution prepared in 3.2.1)]

Y pH of mixed salt solution

Figure A.1

Annex B
(informative)

Typical apparatus for accelerated cyclic tests with exposure to acidified salt spray, “dry” and “wet” conditions



Key

1 cover
2 adjustable baffle
3 test specimen supports
4 test specimen
5 mist-dispersion tower
6 solution
7 pressure gauge
8 compressed air
9 air dryer
10 air saturater
11 air
12 humidifier
13 atomizer
14 water
15 collecting device (remote from an inlet of spray)
16 collecting device (close to an inlet of spray)

Figure B.1