## INTERNATIONAL STANDARD

ISO 11997-1

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# Paints and varnishes — Determination of resistance to cyclic corrosion conditions

Part 1: Wet (salt fog)/dry/humidity

Peintures et vernis Détermination de la résistance aux conditions de corrosion cyclique

Partie 1: Brouillard salin/sécheresse/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 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 11997-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 11997-1:1998) (including Technical Corrigendum ISO 11997-1:1998/Cor.1:1998), in which cycle A has been revised and cycle D has been added to correlate with ISO 14993:2001, Corrosion of metals and alloys — Accelerated testing involving cyclic exposure to salt mist, "dry" and "wet" conditions.

ISO 11997 consists of the following parts, under the general title *Paints and varnishes* — *Determination of resistance to cyclic corrosion conditions*:

- Part 1: Wet (salt fog)/dry/humidity
- Part 2: Wet (salt fog)/dry/humidity/UV light

#### Introduction

Coatings of paints, varnishes and similar materials are exposed to one of four cycles of wet and dry conditions using specified salt solutions in a cabinet in order to simulate, in the laboratory, processes occurring in aggressive outdoor conditions, such as marine environments. Generally, correlation between such outdoor weathering and laboratory testing cannot be expected because of the large number of factors influencing the breakdown process. Correlation can only be expected if the effect on the coating of important parameters (e.g. the nature of the pollutant, the spectral distribution of the incident irradiance in the relevant photochemical region, the temperature of the specimen, the type and cycle of wetting and relative humidity) is known. In contrast to outdoor weathering, laboratory testing in a cabinet is performed with a reduced number of variables which can be controlled, and therefore the effects are more reproducible. The method described may also give a means of checking that the quality of a paint or paint system is being maintained.

The method has been found to be useful in comparing the cyclic salt spray resistance of different coatings. It is most useful in providing relevant ratings for a series of coated panels exhibiting significant differences in cyclic salt spray resistance.

The test cycles included in this part of ISO 11997 have been used successfully, with documented evidence, in the industry for the assessment of performance. The cycles can be summarized as follows.

- Cycle A (see Annex C): This cycle is specified in Japanese Automobile Standards JASO M 609-91, Corrosion test method for automotive materials, and JASO M610-92, Cosmetic corrosion test method for automotive parts.
- Cycle B (see Annex D): This is based on the VDA 621-415 cycle and is widely used in Europe. It has
  also been shown to give good correlation with natural weathering for thermosetting paints in vehicle
  corrosion.
- Cycle C (see Annex E): This cycle was developed in the UK for use with water-soluble and latex paint systems, and has been shown to give good correlation with natural weathering.
- Cycle D (see Annex F): This cycle is specified in Japanese Standard JIS K 5621-2003, Anticorrosive paint for general use.

It is intended that other cycles will be added at later revisions of this part of ISO 11997, as they are developed for evaluating other paint types.

ISO 11997-2 describes a method for determining the cyclic corrosion resistance of paints which includes UV exposure as part of the cycle. It has been found to give good correlation with natural weathering for industrial maintenance coatings.

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## Paints and varnishes — Determination of resistance to cyclic corrosion conditions —

#### Part 1:

#### Wet (salt fog)/dry/humidity

#### 1 Scope

This part of ISO 11997 describes a method for the determination of the resistance of coatings to one of four defined cycles of wet (salt fog)/dry/humidity conditions using specified solutions.

#### 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 1513, Paints and varnishes — Examination and preparation of samples for testing

ISO 1514, Paints and varnishes — Standard panels for testing

ISO 2808, Paints and varnishes — Determination of film thickness

ISO 3270, Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing

ISO 3696, Water for analytical laboratory use — Specification and test methods

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

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

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

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

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

ISO 15528, Paints, varnishes and raw materials for paints and varnishes — Sampling

#### 3 Principle

A coated test panel is exposed to a cyclic wet (salt fog), drying and humidity test schedule and the effects of exposure are evaluated by criteria agreed in advance between the interested parties, these criteria usually being of a subjective nature.

#### 4 Required supplementary information

For any particular application, the test method described in this part of ISO 11997 needs to be completed by the supplementary information given in Annex A.

#### 5 Salt fog test solution

Prepare the salt fog test solution by dissolving the salt or salts as given in Annexes C, De and F in water conforming to at least grade 2 of ISO 3696, to produce the required concentration.

The salts shall be of analytical grade or visually white, and conform with the purity requirements given in Table 1.

Maximum mass fraction of impurity **Impurity** Method of determination % Total 0,5 Calculated as a percentage of the dry salt lodide 0,1 Calculated as a percentage of the dry salt Copper 0,001 Determined by spectrophotometry or another method of similar accuracy Nickel 0.001 Determined by spectrophotometry or another method of similar accuracy

Table 1 — Purity of salts

If the pH of the solution is outside the required range (see Annexes C, D, E and F), the presence of undesirable impurities in the salt or the water or both shall be investigated. The pH determination shall be based on electrometric measurement at 25 °C, but a narrow-range indicator paper which can be read in increments of 0,3 pH-units or less and which has been calibrated against electrometric measurements may be used in routine checks. Any necessary corrections shall be made by adding hydrochloric acid, sodium hydrogen carbonate (both of purity conforming to the requirements of Table 1) or analytical-grade sodium hydroxide, of appropriate concentrations.

NOTE Attention is drawn to the possible changes in pH resulting from loss of carbon dioxide from the solution when it is sprayed, or from dissolution of carbon dioxide from the ambient atmosphere. Such changes may be avoided by reducing the carbon dioxide content of the solution by, for example, heating it to a temperature above 35 °C before it is placed in the cabinet or making the solution from freshly boiled water.

Filter the solution before placing it in the reservoir of the cabinet, in order to remove any solid matter which might block the apertures of the spraying device.

#### 6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

**6.1 Spray cabinet**, made of, or lined with, material resistant to corrosion by the sprayed solution and having a roof which prevents condensed moisture dripping onto the test specimens. The cabinet shall have a capacity of not less than 0,4 m<sup>3</sup> in order to ensure even distribution of the spray.

The size and shape of the cabinet shall be such that the salt fog deposition rate is within the limits given in Annexes C, D, E and F.

NOTE Cabinets with a volume greater than 2 m<sup>3</sup> will be difficult to operate unless careful consideration is given to their design and construction.

Factors to be taken into consideration in the design and construction of the cabinet are given in Annex B.

If the cabinet has been used for a spray test, or for any other purpose, using a solution differing from that specified for the current test cycle, it shall be thoroughly cleaned before use.

- **6.2 Hot-air blowers**, capable of maintaining the cabinet and its contents at the specified temperatures and achieving the required heating rates (see Annexes C, D, E and F). The temperature shall be controlled by a thermostat element placed within the cabinet at least 100 mm from the walls of the cabinet.
- **6.3 Means for spraying the salt solution**, comprising a supply of clean compressed air at a controlled pressure, a reservoir to contain the solution to be sprayed, and one or more atomizers made of material resistant to the solution. The compressed-air supply to each atomizer shall be passed through a filter to remove all traces of oil or solid matter, shall be at a suitable pressure depending upon 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 Annexes C, D, E and F).

The reservoir containing the solution to be sprayed shall be a tank made of material resistant to the solution and shall be provided with means of maintaining a constant flow of solution to the atomizers.

The atomizers shall be made of inert material, for example glass or plastic.

NOTE Baffles may be used to prevent direct impingement of spray on the test specimens, and the use of adjustable baffles is helpful in obtaining uniform distribution of spray throughout the cabinet.

**6.4 Drying-air supply**, of 20 % to 30 % relative humidity for cycles A and D (see Annexes C and F) and of  $(50 \pm 20)$  % relative humidity for cycles B and C (see Annexes D and E). The air shall be passed through a filter to remove all traces of oil or solid matter and shall have a flow rate sufficient to ensure that, at the start of a dry phase, no drops of moisture are visible on the panels after the drying time specified for cycles A and D and after 45 min for cycles B and C. The panels shall not be dried by heaters inside the cabinet walls.

**6.5** Collecting devices, at least two, of chemically inert material (see Note). The collecting devices shall be placed in the zone of the cabinet where the test panels are placed, one close to the spray atomizer(s) and one remote from the spray atomizer(s). They shall be placed so that only spray and not liquid falling from test panels or from parts of the cabinet is collected. If two or more atomizers are used, the number of collecting devices shall be at least twice the number of atomizers.

NOTE Glass funnels with the stems inserted into graduated cylinders have been found to be suitable collecting devices. Funnels with a diameter of 100 mm have a collecting area of approximately 80 cm<sup>2</sup>.

**6.6 Test panel racks**, made of inert non-metallic material such as glass, plastic or suitably coated wood. Exceptionally, test panels may be suspended. In this case, the material used shall be synthetic fibre, cotton thread or other inert insulating material; on no account shall metallic material be used. All racks shall be placed at the same level in the cabinet so that the solution cannot drip from panels or racks at one level onto other panels placed below.

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**6.7 Control equipment**, for providing wet (salt fog)/dry/humidity cycles for the times and temperatures specified in Annexes C, D, E and F.

#### 7 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system), as described in ISO 15528.

Examine and prepare each sample for testing, as described in ISO 1513.

#### 8 Test panels

#### 8.1 Material and dimensions

Unless otherwise specified or agreed, the test panels shall be of burnished steel complying with ISO 1514, and of minimum dimensions  $100 \text{ mm} \times 70 \text{ mm} \times 0.3 \text{ mm}$ .

#### 8.2 Preparation and coating of panels

Unless otherwise specified, prepare each test panel in accordance with SO 1514 and then coat it by the specified method with the product or system under test.

Unless otherwise specified, the back and edges of the panel shall be coated with the product or system under test.

If the coating on the back and edges of the panel differs from that of the product under test, it shall have a corrosion resistance greater than that of the product under test.

#### 8.3 Drying and conditioning

Dry (or stove) and age (if applicable) each coated test panel for the specified time under the specified conditions, and then, unless otherwise specified, condition them at a temperature and relative humidity as defined in ISO 3270 for at least 16 h, with free circulation of air and without exposing them to direct sunlight. The test procedure shall then be carried out as soon as possible.

#### 8.4 Thickness of coating

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

#### 8.5 Preparation of scribe

Unless otherwise specified, make a straight scribe with a minimum width of 0,2 mm through the coating to the substrate as follows:

To make the scribe, use a single-blade cutting tool. The scribe shall have an upwards-broadening cross-section which shows a width of 0,2 mm to 1,0 mm of the metallic substrate. Remove any loose debris from the vicinity of the scribe.

Do not cut the scribe with a knife.

For aluminium panels, make two scribes which are perpendicular to, but do not intersect, each other. One scribe shall be parallel to the rolling direction and the other at right angles to it.

All scribes shall be at least 20 mm from each other and from any edge of the test panel, unless agreed otherwise.

Two parallel scribes may also be applied. Unless agreed otherwise, locate the scribes parallel to the longer edge of the test panel.

Care needs to be taken in the production of the scribe to ensure uniformity of results.

#### 9 Method of exposure of test panels

Place the panels in the cabinet in such a way that they are not in the path of spray from the atomizer.

Place the panels facing upwards at an angle of  $(20 \pm 5)^{\circ}$  to the vertical.

The angle at which each panel is exposed in the cabinet is very important.

It is sometimes necessary to expose painted components of different shapes. When such tests are conducted, it is of particular importance to expose the shaped components at the angle at which they are normally used. While satisfying this restriction, the component shall also be placed so as to minimize the disruption of flow. Furthermore, other test panels and components cannot be tested at the same time if the shape of the painted component interferes with the general direction of flow.

The degree of film breakdown may vary with different test specimen orientations, and due consideration shall be given to this when interpreting the results.

Arrange the panels so that they do not come into contact with one another or with the cabinet and so that the surfaces to be tested are exposed to the spray only where it is settling freely.

It is recommended that the positions of the test panels be rotated on a daily basis to ensure positional effects within the cabinet do not interfere with the degradation process.

#### 10 Operating conditions

Set the cabinet to run on the required spray cycle only and determine the salt fog deposition rate. The average rate of collection of solution in each collecting device (6.5) measured over a minimum period of 24 h shall be as given in the appropriate annex (C, D, E or F) for a horizontal collecting area of 80 cm<sup>2</sup>. This gives a more accurate assessment of the deposition rate, as the water will evaporate away from the collection devices during the drying cycles.

The solution collected shall have the concentration given in the appropriate annex (C, D, E or F).

Operate the cabinet to the required cycle (see Annex C, D, E or F) unless otherwise specified, repeating the cycle for the required time.

Do not re-use test solution which has already been sprayed.

#### 11 Procedure

Carry out the determination in duplicate, unless otherwise agreed.

Set up the cabinet as described in Clause 10 and allow equilibrium to be established.

Arrange the test panels in the cabinet (6.1) as described in Clause 9.

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It is recommended that a control specimen of a paint with known durability be included with each series of test panels and that duplicates be exposed in different parts of the chamber.

Close the cabinet and start the flow of the salt solution through the atomizers. Run the cycle continuously throughout the prescribed test period, except for a short daily interruption (see Clause 12) to inspect, re-arrange or remove test panels, to check the atomizers, to check and replenish the solution in the reservoir and to make any necessary recordings.

#### 12 Examination of test panels

Make a periodic examination in accordance with the criteria agreed in advance between the interested parties.

Examine the panels as quickly as possible, taking care not to damage the surfaces under test. Do not turn off the cabinet for more than 30 min in any 24 h period. Examine the panels during a dry phase. If this is not possible for any reason, turn off the cabinet but do not allow the panels to dry, and restart the cabinet as soon as possible.

At the end of the specified test period, remove the panels from the cabinet and rinse with clean water to remove residues of salt solution from the surface. Immediately examine the test surfaces for signs of deterioration in accordance with ISO 4628, Parts 1 to 5 [see Annex A, item g)].

If required, keep the panels in a standard atmosphere as defined in ISO3270 for the specified period and re-examine the test surfaces for deterioration.

If it is required to examine the substrate for signs of attack, remove the coating by means of a non-corrosive paint remover unless otherwise specified [see Annex A, item g)].

#### 13 Precision

The concept of precision is not applicable to this part of ISO 11997 because of the subjective nature of the assessment (see Clause 3). Precision depends upon a number of factors, including:

- the evaluation method (i.e. ISO 4628, Rarts 1 to 5);
- the preparation of the test panels
- the thickness of the coating
- the drying and conditioning of the test panels;
- the preparation of the scribe(s).

#### 14 Test report

The test report shall contain at least the following information:

- a) all information necessary to identify the product tested;
- a reference to this part of ISO 11997 (ISO 11997-1:2005);
- c) the cycle used;
- d) the items of supplementary information referred to in Annex A;

- a reference to the international or national standard, product specification or other document supplying the information referred to in d) above;
- the duration of the test; f)
- the scribe instrument used; g)
- the length, cross-sectional dimensions and location of each scribe mark; h)
- i) whether the position of the panels was changed (see Clause 9);
- STANDARDSISO COM. Click to view the full POF of 150 1, 1997 A. 2005 j)
- k)
- I)
- m) the date of the test.

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## Annex A

(normative)

#### Required supplementary information

The following items of supplementary information shall be supplied as appropriate to enable the determination to be carried out. The information required should be agreed between the interested parties and may be derived, in part or totally, from an international or national standard or other document related to the product under test.

- The material, dimensions and surface preparation of the substrate (see 8.1 and 8.2).
- The method of application of the test coating to the substrate (see 8.2). b)
- The duration and conditions of drying (or stoving) and ageing (if applicable) of the coated test panels before testing (see 8.3).
- The thickness, in micrometres, of the dry coating and method of measurement in accordance with ISO 2808, and whether it is a single coating or a multi-coat system (see 8.4).
- e) Whether a scribe is to be made before exposure and, if so, the type of scribing tool, the depth and width of each scribe, its position and any other required information (see 8.5).
- The duration of the test (see Clause 10). f)
- How inspection of the test coating is to be made and what characteristics are to be considered in STANDARDSISO.COM. Click's evaluating its resistance to wet (salt fog)/dry/humidity (see Clause 12).
- The cycle to be used.

### Annex B

(informative)

#### Factors to be considered in the design and construction of salt spray cabinets

The following factors should be considered in the design and construction of the cabinet (see 6.1):

- the number and the positioning of the atomizers and baffles required to produce an even spray distribution within the limits specified in Annex C, D, E or F;
- b) the number of collecting devices required to monitor the spray as required in 6.5;
- c) the heating, insulation and means of temperature control required to produce a uniform temperature within the cabinet at all positions where test specimens are exposed;
- d to prev. d. to prev. circk to view the full prof. of STANDARDS ISO. COM. d) the design of the ceiling (for example a false ceiling) needed to prevent drops of solution falling onto the

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#### Annex C (normative)

#### Cycle A

#### C.1 Salt fog test solution

71997.7:2004 Prepare the spray solution as described in Clause 5 by dissolving sodium chloride in water to give a concentration of (50  $\pm$  10) g/l. The pH of the solution shall be in the range 6,0 to 7,0.

#### C.2 Cycle

Set up the cabinet to perform the following cycle:

Step	<b>Time</b> h	Temp. °C	Condition	Notes	
1	2	35 ± 2	Salt spray		
2	4	60 ± 2	Dry: 20 % to 30 % RH		
3	2	50 ± 2	Wet: 95 % RH or over	60.	
4	4 Go back to step 1			This makes a total cycle time of 8 h from step 1 to step 3.	
	a in			From salt spray to dry: within 30 min	
				From dry to wet: within 15 min	
	Transition		time allowed to reach the tive humidity specified for a		
	condition after changing to that condition):			The effect of the salt spray will, in principle, be instantaneous.	
				For details of drying-air flow, see 6.4.	

NOTE Set points and operational fluctuations can be listed either independently of each other or in the format: set point ± operational fluctuations. The set point is the target condition for the sensor when used at the operational control point as programmed by the user. Operational fluctuations are deviations from the indicated set point at the control point indicated by the readout of the calibrated control sensor during equilibrium operation and do not include measurement uncertainty. At the operational control point, the operational fluctuation shall not exceed the listed value at equilibrium. When a standard calls for a particular set point, the user programmes that exact number. The operational fluctuations specified for that set point do not imply that the user is allowed to programme a set point higher or lower than the exact set point specified.

#### C.3 Salt fog deposition rate

The salt fog deposition rate shall be 1 ml/h to 2 ml/h, when the salt fog is collected over a 24 h period (see Clause 10).

#### C.4 Duration

Unless otherwise agreed, repeat the test cycle for 30 cycles (240 h), 60 cycles (480 h), 90 cycles (720 h) or 180 cycles (1 440 h).