
**Paints and varnishes — Determination of
resistance to cathodic disbonding of
coatings exposed to sea water**

*Peintures et vernis — Détermination de la résistance au décollement
cathodique des revêtements exposés à l'eau de mer*

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Published in Switzerland

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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 15711 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

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Introduction

This International Standard describes two methods for determining the ability of paint or other organic coatings applied to metallic substrates to withstand cathodic disbonding. These methods may also afford a basis for the comparison of particular coatings. The conditions of test are more severe than those likely to be encountered normally and so coating failure may be accelerated. It is recommended that the test be carried out for a period of at least 26 weeks and hence these methods are not suitable as a means of achieving quality control.

The methods are suitable for coatings used for the protection of ships and structures exposed to sea water. Method A is based on the procedure developed and evaluated by COIPM (Comité International Permanent pour la Protection des Matériaux en Milieu Marin).

The test result may be influenced not only by the properties of the coating system under test, but also by the nature and preparation of the substrate, the method of application of the coating system and other factors.

Paints and varnishes — Determination of resistance to cathodic disbonding of coatings exposed to sea water

1 Scope

This International Standard describes two methods for determining the ability of paint, or other organic coatings, applied to metallic substrates to withstand cathodic disbonding when the surface coating may contain or develop discontinuities. The methods are applicable to coatings that are exposed to sea water, such as those applied to ships or marine structures. They are not suitable for the assessment of the ability of coatings to withstand cathodic disbonding on land-based structures.

Method A involves the use of a cathodic protection circuit, whereby the electrode potential of the test substrates is controlled potentiostatically.

Method B involves the use of sacrificial anodes attached to the test substrates. This reflects the practical method of cathodic protection commonly used on ships.

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 554, *Standard atmospheres for conditioning and/or testing — Specifications*

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 method*

ISO 4624, *Paints and varnishes — Pull-off test for adhesion*

ISO 4628-2, *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 8501-1, *Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness — Part 1: Rust grades and preparation grades of uncoated steel substrates and of steel substrates after overall removal of previous coatings*

ISO 12944-6, *Paints and varnishes — Corrosion protection of steel structures by protective paint systems — Part 6: Laboratory performance test methods*

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

3 Terms and definitions

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

3.1 cathodic disbonding
failure of adhesion between a coating and a metallic surface that is directly attributable to cathodic protection conditions and that is often initiated by a defect in the coating system, such as accidental damage, imperfect application or excessive permeability of the coating

4 Principle

4.1 Method A (impressed current)

A coated test panel is damaged in a defined way. The panel is then exposed in a test tank and connected to a cathodic protection circuit. The effects of exposure are evaluated against criteria which correspond to ISO 12944-6 (e.g. ISO 4628-2 or ISO 4624).

4.2 Method B (sacrificial anode)

A sacrificial metal anode is attached electrically to a metal substrate which has been coated with the test coating/system. The inherent electrochemical nature of the two metals coupled in this manner establishes the test potential.

The coating under test is applied to four panels in total. Two of these panels are protected by the sacrificial metal anodes and the other two, unprotected, panels act as controls. Artificial defects are introduced on all four test panels which are then immersed for six months in sea water.

After the immersion period, the test panels are removed from the sea water and immediately examined for blister formation and creep from the defect. An assessment of the coating's performance under cathodic protection conditions is made by comparing the results from the protected and unprotected panels.

5 Required supplementary information

For any particular application, the test method specified in this International Standard needs to be completed by supplementary information. The items of supplementary information are given in Annex A.

6 Test solutions

6.1 Artificial sea water, conforming to the requirements given in Table 1, shall be used unless otherwise agreed (see 6.2).

Table 1 — Artificial sea water composition

Compound	Concentration
	g/l
Sodium chloride	23,0
Magnesium chloride hexahydrate	9,8
Sodium sulfate decahydrate	8,9
Calcium chloride	1,2

The formulation shall contain no other additives, e.g. EDTA.

Use water conforming as a minimum to the requirements of grade 2 of ISO 3696, and chemical reagents of analytical grade, for all solutions.

6.2 Natural sea water may be used if specified. If necessary, suspended solids may be removed by filtering through a suitable filter paper.

7 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

7.1 Method A (impressed current)

7.1.1 Test tank, of glass or other inert material that will not affect the pH of the electrolyte (6.1 or 6.2). The tank, either round of diameter not less than 700 mm or rectangular of minimum side 700 mm, shall have a depth of not less than 200 mm. A system for aerating the electrolyte shall be provided.

7.1.2 Anode, of graphite, with a minimum length of 200 mm and a minimum diameter of 10 mm, or of platinum wire, platinum gauze, platinized titanium or other materials which show sufficient resistance to anodic polarization in sea water.

7.1.3 Reference electrode, such as a saturated calomel or Ag/AgCl electrode, constructed from either glass or plastic, with a porous plug. If an Ag/AgCl or other reference electrode is used, it shall be checked against the saturated calomel electrode and measured potentials corrected in accordance with Table 2.

Table 2 — Potentials of reference electrodes with respect to the standard hydrogen electrode (at 25 °C)

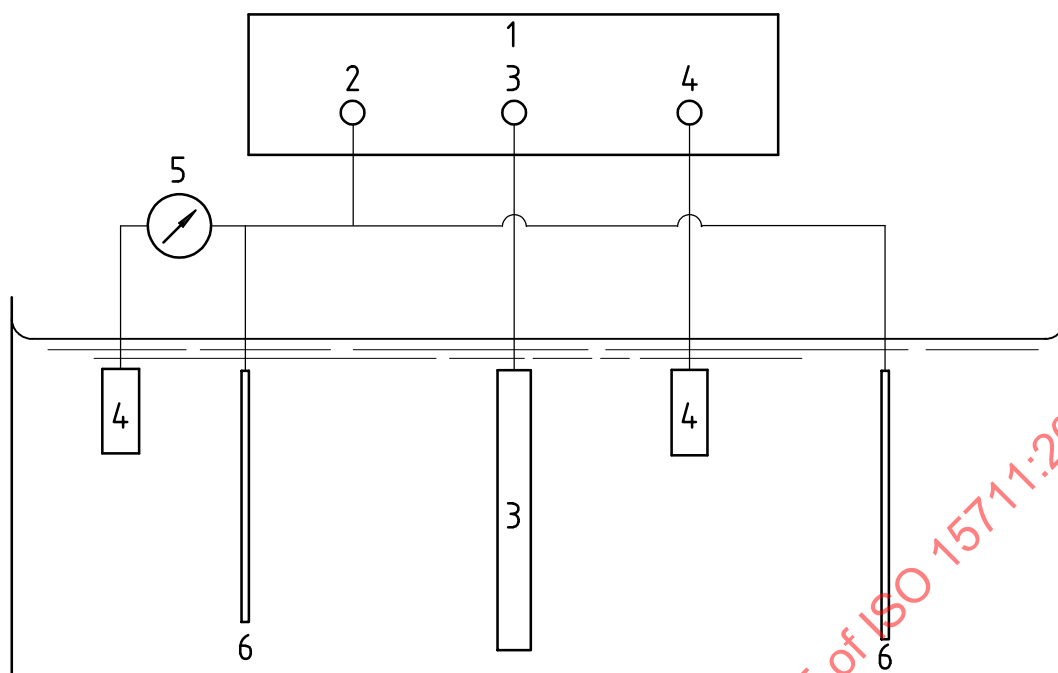
Electrode	Potential V
Copper/copper sulfate	+ 0,32
Silver/silver chloride/saturated KCl	+ 0,20
Silver/silver chloride/sea water	+ 0,25 (approx.)
Calomel (normal KCl)	+ 0,28
Calomel (saturated KCl)	+ 0,25

7.1.4 Voltmeter, with a minimum resistance of 10 M Ω and capable of measuring in the range 0 V to 2 V to the nearest 1 mV.

7.1.5 Cathodic protection circuit, operating from a d.c. supply and capable of maintaining the electrode potential of the test substrate at $(-1\,050 \pm 5)$ mV relative to the saturated calomel reference electrode.

The potential should preferably be controlled potentiostatically, but if a potentiostat is not available then a galvanostat may be used. The potential shall be frequently checked and adjusted to ensure that the electrode potential is within the specified limits (see 10.3.1).

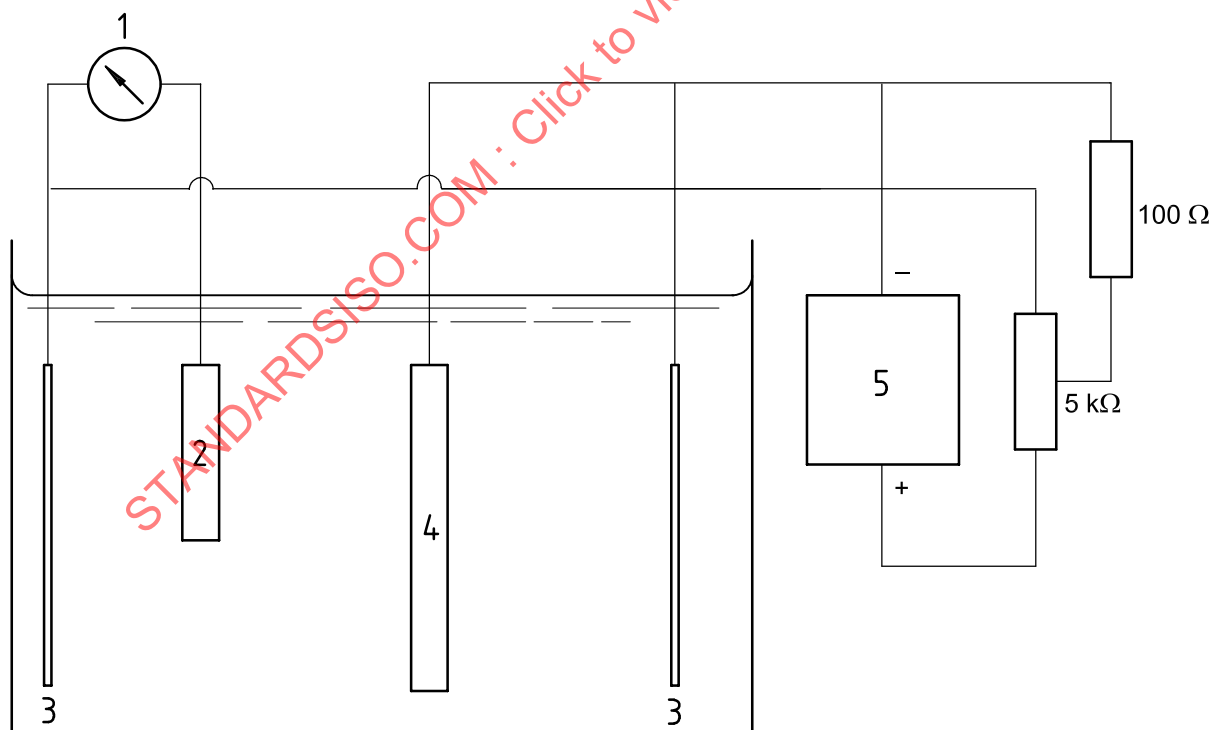
Typical cathodic protection circuits are shown in Figures 1 and 2.



Key

- | | |
|---------------------|--------------------------|
| 1 potentiostat | 4 reference electrode(s) |
| 2 working electrode | 5 voltmeter |
| 3 anode | 6 test panel |

Figure 1 — Potentiostatically controlled cathodic protection circuit



Key

- | | |
|-----------------------|---------------|
| 1 voltmeter | 4 anode |
| 2 reference electrode | 5 d.c. source |
| 3 test panel | |

Figure 2 — Galvanostatically controlled cathodic protection circuit

7.2 Method B (sacrificial anode)

7.2.1 Test tanks, made of glass or reinforced plastic, with means for thermostatic temperature control and water circulation/aeration. The capacity of the tanks shall be such that they contain not less than 7,5 l of water per 1 m² of panel surface exposed (if the panel spacing specified in 7.2.2 is adopted, the water:panel-area ratio well exceeds this figure). The tanks shall be of a depth such that panels 300 mm in height can be fully immersed with 50 mm of water above the top of the panel.

7.2.2 Test panel racks, non-electrically conductive, to hold panels at no more than 20° to the vertical, at a minimum panel separation of 30 mm, with 50 mm minimum clearance from the tank walls and insulated from the tank bottom. Spacing shall be such that the panels do not come into contact with each other.

7.2.3 Standard drill press and milling cutter/slot drill bit, of 3 mm diameter, for introduction of artificial defects (see 9.5.2).

7.2.4 Electromagnetic dry film thickness meter.

7.2.5 Penknife, for adhesion testing. A knife with a 5 cm to 6 cm blade has been found to be suitable.

7.2.6 Panel edging "tray", of such dimensions as to enable the panel edges to be dipped in along their full length.

7.2.7 Zinc anodes, having a composition as described in Annex B, to provide a potential of $(-1\,040 \pm 10)$ mV relative to a saturated calomel reference electrode. Each anode shall have a hole drilled through the centre (of such diameter as to enable attachment to the steel bolt which has been previously welded to the test panel) and be of approximate dimensions 50 mm × 40 mm × 10 mm, to give a mass of (125 ± 5) g.

If pre-used anodes are to be used, in order to ensure that they are active they shall be cleaned by wire brushing or by immersion in dilute acid to remove corrosion products.

7.2.8 Serrated galvanized mild-steel locking washers and galvanized mild-steel nuts.

7.2.9 pH-monitoring equipment: a pH-meter or pH-papers.

7.2.10 Temperature-monitoring equipment: a thermometer or data-logging device.

8 Sampling

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

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

9 Test panels

9.1 Material and dimensions

9.1.1 Method A (impressed current)

Unless otherwise specified, the test panels shall be of the same material and prepared to the same standard as the substrate to which the coating will be applied.

If such test panels are not available, steel test panels conforming to the requirements of ISO 1514 shall be used, prepared in the same manner as the representative substrate.

The test panels shall be of minimum dimensions 150 mm × 70 mm × 2 mm, unless otherwise specified.

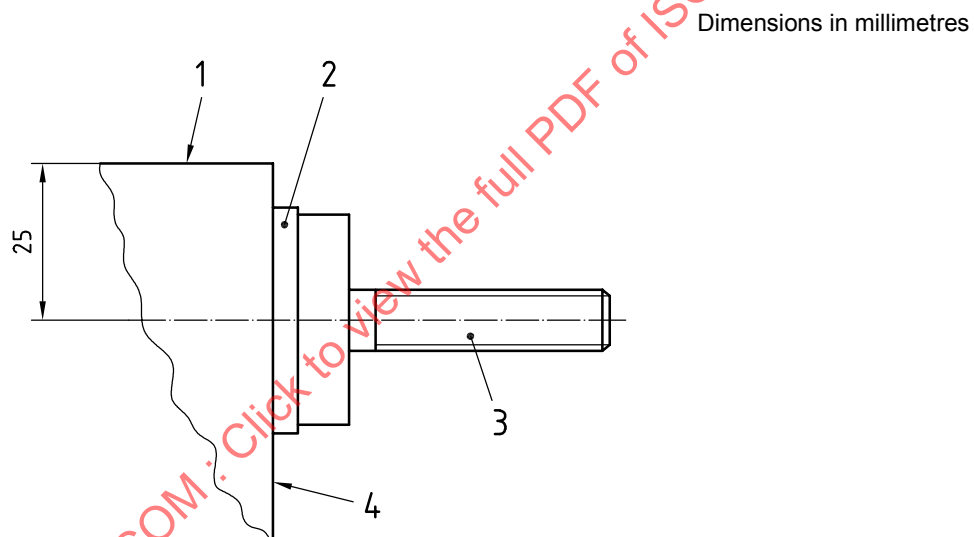
Each test panel shall have an insulated conductor, firmly attached by a self-tapping screw, or by a small nut and bolt, or by soldering, or by a clip, or by any other means which gives a sound electrical connection which is not affected by corrosion.

9.1.2 Method B (sacrificial anode)

Unless otherwise specified, the test panels shall be of the same material, and prepared to the same standard, as the substrate to which the coating will be applied in practice.

For steel substrates, four panels each measuring 300 mm × 150 mm × 2 mm are required for each coating or coating system under test, i.e. two for testing under cathodic protection and two unprotected controls. If any other panel dimension is used, the area of the introduced defect shall be scaled accordingly to provide the same test-area:defect-area ratio, remembering that the test area includes both sides of the panel.

Spot-weld a galvanized mild-steel bolt onto one of the longer edges of two of the mild-steel panels, as shown in Figure 3.

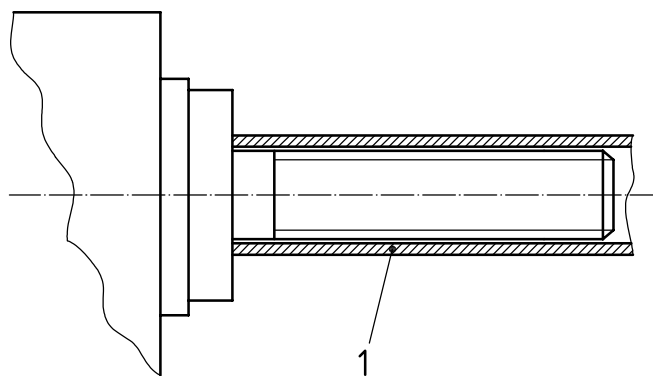


Key

- 1 mild-steel panel
- 2 spot weld
- 3 galvanized mild-steel bolt
- 4 longer edge of panel

Figure 3 — Attachment of galvanized mild-steel bolt to test panel (method B)

Place a small piece of solvent-resistant plastic tubing over the screw threads to protect them during surface preparation and coating, as shown in Figure 4.



Key

1 plastic protector

Figure 4 — Protection of bolt prior to coating (method B)

9.2 Preparation and coating

9.2.1 Method A (impressed current)

Prepare the test panels to the same standard as the substrate which will normally be used (see 9.1.1). If no treatment is specified, prepare them by blast-cleaning as described in ISO 1514, and then coat them by the specified method with the product or system under test. Coat the back and edges of the panel with the product or system under test, unless otherwise specified. Coat the edges carefully with a film thicker than that specified in order to avoid edge effects.

Protect any uncoated surfaces of the test substrate and the connection to the insulated conductor with a coating of paraffin wax applied at a temperature of 70 °C or a solventless epoxy resin or other suitable coating which has better protective properties than the material under test.

Also prepare three control panels which are not to be connected to the cathodic control system.

9.2.2 Method B (sacrificial anode)

Unless otherwise specified, thoroughly degrease all four panels and grit-blast to a near-white metal finish (Sa 21/2 as defined in ISO 8501-1). It is recommended that G24 iron grit be used to give a blast profile of between 50 µm and 100 µm, as creep is a surface area effect and can therefore be influenced by the blast profile. Hence, standardization of the grade of grit used reduces variability between test laboratories.

Apply the test coating/system to both sides of the four panels, using the recommended method of application (airless spray, conventional spray, brush or roller). When there are only small quantities of paint available, apply the coating/system by brush.

In all cases, apply the same coating system, of the same film thickness, to each side of the panel. The reverse of the panel is subjected to the same exposure conditions and therefore the performance of the coating on the reverse of the panel will directly affect that of the coating on the front.

It is recommended that an appropriate product or system of known performance is included in the test programme as a “benchmark”.

Before the preliminary drying period is completed, carefully edge the panels with the same paint as that under test. Take care not to damage the test area when allowing the edging paint to dry.

9.3 Drying and conditioning

Dry (or stove) and age (if specified) each coated test panel for the specified time under the specified conditions and, unless otherwise specified, condition them at one of the standard temperatures and relative humidities defined in ISO 3270 or ISO 554 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.

9.4 Thickness of coating

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

9.5 Formation of an artificial holiday

9.5.1 Method A (impressed current)

Immediately before carrying out the test, introduce an artificial holiday into the coated surface of each of the test panels, using the following preferred method unless otherwise specified. Place a blast-resistant mask with a cut-out hole of (10 ± 1) mm diameter not less than 30 mm from any edge on the surface and blast-clean the panel using the procedure defined in ISO 1514 until the coating has been completely removed from the exposed area. The artificial holiday may also be prepared by milling, but the results may differ from that of the preferred method.

Check that an electrical connection exists between the holiday and the conductor.

9.5.2 Method B (sacrificial anode)

Immediately prior to immersion, introduce a circular artificial defect into the centre of the front face of all four panels, preferably using an approximately 3 mm diameter flat-ended drill bit (7.2.3).

Take care to remove the coating/system and only the immediate steel surface profile to leave a uniformly smooth surface. Excessive metal removal may introduce localized heating that could either damage the surrounding coating or change the electrochemical potential of the adjacent steel.

Introduce the defect on all four panels, in the centre of the test area, and remove all paint and steel debris with a soft brush prior to immersion. It is recommended that a clamp be used to hold the panel whilst the defect is being introduced to ensure uniform location of the defect and also to prevent panel/cutter slippage.

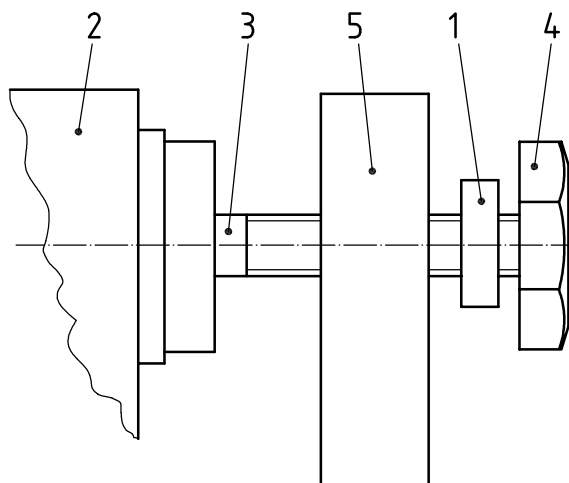
If the artificial defect is introduced by a method other than that recommended above, the results may differ from those of the preferred method.

9.6 Presence of accidental holidays

Examine the test panel for the presence of accidental holidays by means of a flaw detector fitted with a straight, soft, metal-bristle brush approximately 75 mm long and 6 mm wide with bristles 6 mm long, adjusted to provide a voltage of 10 kV but so arranged that the maximum short-circuit current is not greater than 8 mA. If the flaw detector is not equipped with a voltage indicator, set it to give a 12 mm spark in air to a steel plate. Pass the flaw detector over the test panel at a distance of approximately 6 mm from the steel surface. Ignore any edge effects.

9.7 Attachment of anode (method B only)

Remove the protective tubing from the screw thread. Ensure the thread is clean and then attach the metal anode using a serrated galvanized mild-steel washer and nut as shown in Figure 5. Tighten the nut using a spanner until the anode is secure.



Key

- 1 serrated galvanized mild-steel washer
- 2 test panel
- 3 galvanized mild-steel bolt
- 4 galvanized mild-steel nut
- 5 anode

Figure 5 — Attachment of anode (method B)

Zinc alloy anodes are most commonly used in the marine industry, of a composition as described in Annex B. These anodes provide a potential of $(-1\,040 \pm 10)$ mV relative to a saturated calomel reference electrode. Other metals may be used, but will produce a different potential across the test panel and will therefore give different results.

10 Procedure

10.1 Number of tests

For method A, carry out all tests in triplicate and for method B in duplicate unless otherwise agreed.

10.2 Arrangement of test panels in the tank

10.2.1 Method A (impressed current)

Place the anode (7.1.2) in the centre of the tank (7.1.1) and connect it to the positive terminal of the cathodic protection circuit (7.1.5).

Label each panel with a unique identifier, using a suitable material which will not fade during the test. Locate the test panels, prepared as specified in Clause 9, in racks in the tank. Adjust their position such that each test panel is not less than 300 mm from the anode (7.1.2) in the tank, is not less than 50 mm from the base, and will be totally immersed when the tank is filled with the electrolyte. Ensure that no test panel is touching another and that no test panel is in contact with the side of the tank. Ensure that no panel face containing a holiday is shielded from the anode by any obstruction, including another test panel, and that the flow of electrolyte around and between the test panels is not impeded.

Connect each test panel to the negative terminal of the cathodic protection circuit by means of the insulated conductor.

Locate the control panels in a similar manner in a separate, equivalent, tank without the anode and cathodic protection circuit.

10.2.2 Method B (sacrificial anode)

Label each panel with a unique identifier, using a suitable material which will not fade during the test. Immerse cathodically protected and unprotected control panels in separate, dedicated, tanks under the same conditions. This will prevent possible build-up of zinc ions which would affect the corrosion process on the unprotected panels.

Support the panels in racks (7.2.2) to avoid the need for drilling holes through the panels for suspension purposes. Uncontrolled metal exposure at such holes results in an unquantifiable variable within the test.

It is important that the panels are fully immersed in the electrolyte. When a panel is only partially immersed, there are two distinct corrosion processes which take place, one below and one at the waterline where there is increased availability of oxygen. To avoid the problem of trying to differentiate between the two processes, immerse the panels, with the anode at the bottom in the case of the cathodically protected panels, such that the water level is at least 50 mm above the top of the panel.

10.3 Conditions

10.3.1 Method A (impressed current)

Fill the tank to a minimum depth of 200 mm with the electrolyte (6.1 or 6.2). Commence aeration and check that the electrolyte is at $(23 \pm 2) ^\circ\text{C}$, unless otherwise specified. If the electrolyte used is natural sea water (6.2) and it is not feasible to adjust its temperature, then monitor the temperature of the electrolyte in the tank at appropriate regular intervals and record the readings in the test report.

Maintain a continual flow of the electrolyte through the tank by introducing fresh, aerated electrolyte at the base and allowing it to overflow. Adjust the rate of flow so that the electrolyte is completely replaced in the tank in a maximum of 3 days. Alternatively, replace the electrolyte totally at intervals not exceeding 7 days, and report the procedure used and the frequency of replacement of the electrolyte in the test report.

Switch on the cathodic protection circuit and adjust the current by means of the controlling potentiostat so that each test panel is maintained at an electrode potential of $(-1\,050 \pm 5) \text{ mV}$ with reference to the reference electrode (7.1.3). Check the potential at least once every 24 h and adjust if necessary.

NOTE 1 If the current is monitored for any sudden changes, this may indicate failure of the system and the test may be terminated early.

Check the temperature of the electrolyte daily and adjust if necessary.

Unless otherwise agreed, continue the test for a period of at least 26 weeks.

NOTE 2 The minimum test time is 26 weeks, but the test may be carried out for longer.

10.3.2 Method B (sacrificial anode)

Aerate the test solution and maintain at a constant temperature of $(23 \pm 2) ^\circ\text{C}$, unless otherwise specified. Check the temperature of the test solution daily and adjust if necessary.

Maintain a continual flow of the electrolyte through the tank by introducing fresh, aerated electrolyte at the base and allowing it to overflow. Adjust the rate of flow so that the electrolyte is completely replaced in the tank in a maximum of 3 days. Alternatively, replace the electrolyte totally at intervals not exceeding 28 days, and report the procedure used and the frequency of replacement of the electrolyte in the test report.

Unless otherwise specified, continue the test for a period of 26 weeks.

10.4 Inspection and assessment of the coating

10.4.1 General

Inspect the test and control panels non-destructively at any intermediate stages and on completion of the test as specified.

10.4.2 Intermediate inspections

At intermediate stages, remove each panel in turn and quickly rinse with tap water, taking care not to damage the coating or remove any calcareous deposit at the holiday. Remove the panel for the minimum time possible. Do not allow the panel to dry out.

As alkali may be formed beneath the coating during the test, take care when handling the test panels. While rinsing may reduce the alkali concentration beneath a blister, the concentration may still be sufficient to cause skin burns.

Examine the coating generally, including the back of the panel if this was also coated with the product under test, for signs of blistering (the coating is examined for loss of adhesion in the region of the holiday at the end of the test period only). Record the degree of blistering according to the rating scales described in ISO 4628-2 and the distance of the blisters from the holiday. Record if the coating is peeling away from the substrate at the holiday.

Compare the results obtained for the test coatings with those obtained for the control coatings. Also compare the results with any standard product included in the test.

After the examination, replace the panels in the tank, reconnect and continue the test.

10.4.3 Inspection at end of test

On completion of the test, thoroughly rinse each panel with tap water, taking care not to damage the coating.

Examine the coating generally, including the back of the panel if this was also coated with the product under test, for signs of blistering. Record the degree of blistering according to the rating scales described in ISO 4628-2 and the distance of the blisters from the holiday. It is important to differentiate between blisters associated with the defect and those located away from the defect.

Compare the location of blisters against the original dry film thickness measurements to ascertain if the blisters are associated with areas of low film thickness.

Assess loss of adhesion at the artificial holiday by using a sharp knife to make two cuts through the coating to the substrate, intersecting at the holiday. With the point of the knife, attempt to lift and peel back the coating from around the holiday. Record whether the adhesion of the coating to the substrate has been reduced and the approximate distance, in millimetres, that the coating can be peeled.

Compare the results obtained for the test coatings with those obtained for the control coatings. Also compare the results with any standard product included in the test.

11 Expression of results

Report the results of all examinations of all panels tested.