

Training document CD-Tester

Determination of the resistance against cathodic delamination of coatings in sea water according to the following norms/ standards/ guidelines:

- **ASTM G-8**
- **ASTM G-42**
- **DIN 30 670**
- **DIN EN ISO 15711**
- **ISO 21809 Part 1 Annex H Bestimmung des Widerstandes gegen kathodische Enthftung von Beschichtungen im Meerwasser nach folgenden Normen/ Standards/ Richtlinien:**

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1. Introduction

1.1. General information

There are various methods for the examination of paints and varnishes, generally referred to as coatings and coating materials, which are usually supported by a corresponding set of regulations.

In the present case it is a matter of the so-called cathodic delamination of paints in areas that show damage. The area of application for these components is typically in seawater environments.

So-called cathodic delamination occurs in connection with the cathodic protection of components. The aim of cathodic protection is to further protect components whose coating has been damaged by mechanical influence against corrosion. Such a cathodic protection can be realized e.g. by a sacrificial anode or also by an external current source.

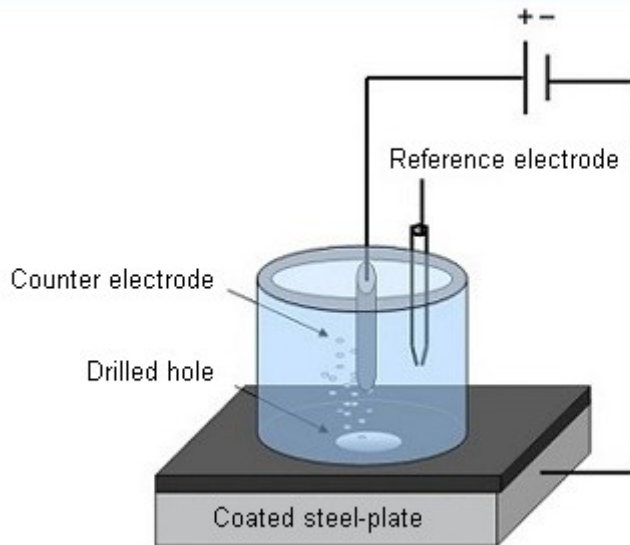
1.2. Utilization

Testing the adhesion of plastic coatings on steel.

1.3. Characteristics

The coating of a sample is damaged in a defined way, so that the steel substrate protrudes. This sample is then cathodically polarized with respect to an anode, i.e. the steel is connected to a controllable current source at the negative pole (the steel sample is overloaded with electrons). After the test, the coating is removed and the extent of infiltration is evaluated.

1.4. Description of the method



The cathodic disbondment test (CD test) examines the susceptibility of coatings to delamination at a defect caused by KKS. The test also provides information about the quality of a coating, since surface preparation and application have an influence on cathodic infiltration.

The test for cathodic disbonding (CD) simulates the condition that occurs at a leakage point either through cathodic protection currents or through hydrogen formation. The coating of a sample is damaged in a defined manner so that the steel substrate is bare. This sample is then cathodically polarized with respect to an anode, i.e. the steel is connected to an adjustable current source at the negative pole.

2. Performance of the test



2.1. Components

To carry out the test, first of all the completeness of all components must be checked. These are as follows:

- Sample: (Steel plate with a defined damage, typically a defect that was created with a 6mm drill. The flaw should be in the middle of the plate). To contact the potentiostat, the sample must have a further damage at the edge or a 4mm hole. A banana plug can be inserted directly into the 4mm hole. If the lacquer is scratched away, a crocodile clip can be used for contacting.

- Measuring cell consisting of:
 - Glass cylinder
 - Lid
 - Counter electrode
 - Reference electrode
 - Intermediate vessel for the reference electrode
 - Silicone with syringe to fix the glass cylinder on the sample
 - 3mol KCL as buffer solution for the intermediate vessel

- External current source, typically a potentiostat, in this case a **Jaissle P-M100**

	
Potentiostat P-M100 with 2 modules for two parallel experiments	CD cell with counter electrode, intermediate vessel and reference electrode

2.2. Execution

Start by applying a thin layer of silicone to the bottom of the glass cylinder.

Then position the glass cylinder with the silicone side centered over the damage in the paint on the sample (steel plate to be tested).

Afterwards you have to wait a certain time until the silicone is solid, otherwise the electrolyte to be filled in later will be pressed outwards.

When the silicone is dried, place the lid on top of the glass cylinder.

Now fill the electrolyte into the cell container through one of the two openings. The electrolyte should have the same composition as sea water. The exact specifications can be found in the respective DIN standard. Add 23.0g sodium chloride, 9.8g magnesium chloride hexahydrate, 8.9g sodium sulfate decahydrate and 1.2g calcium chloride to one liter of distilled water.

Now you can insert the counter electrode (platinum-plated titanium rod with red plug head) and the intermediate vessel (glass cylinder with red screw cap) into the cell cover.

Fill the intermediate vessel approximately halfway with a saturated, but at least with a 3 molar NaCl solution. For a 3 molar NaCl solution we need 3 x 58.5g NaCl per litre distilled water.

A bottle with a volume of 0.5 litres with 87.8g NaCl is supplied. Fill this bottle with distilled water, close it and shake well until all salt crystals have dissolved. Then it can be used as a buffer solution for filling the intermediate vessels.



If the intermediate vessel is filled, the reference electrode (the part with the black head) can also be inserted into the intermediate vessel. To do this, loosen the red cover, push the reference electrodes almost completely into the opening of the seal (approx. 1 cm of the white cylinder can remain outside) and then tighten the cover slightly to fix the electrode.

Now the potentiostat can be connected:

Connect

1. the right black banana socket with the sample (steel plate).
2. the reference electrode with the blue banana socket.
3. the counter electrode (red plug head) with the red banana socket.

If everything is connected correctly, you can now read the so-called OCP on the left instrument of the potentiostat. It is the voltage difference between the steel plate and the reference electrode. The value of this potential depends on the material of the steel plate and on the electrolyte solution. Depending on the material or composition, different potentials can be set.

The value of the potential will drift slightly at the beginning of the test and should then remain at a stable value for the most part. The duration of the drift process can be a few seconds, minutes or even hours. For this test it is not important to wait for a stable state to be reached.

It is important that the potential value is either stable or drifts slightly. If the value hops, then something is wrong with the structure.

Errors can occur:

- Poor contact on the sample (contamination at the contact point)
- Reference electrode is not immersed (too little buffer solution in the intermediate vessel)
- Electrodes not properly connected

If everything is in accordance, the potential setpoint for the test can be set.

Depending on the regulations on which the test is based, these can be different potential values. A typical value is -1260mV against a hydrogen electrode.

Explanation:

In electrochemistry, the so-called hydrogen potential is considered to be the reference potential with the value of 0V. All measurements are converted to this potential unless a hydrogen reference electrode is used.

Since it is slightly cumbersome to establish a hydrogen reference potential, second-order electrodes are typically used in practice. In the majority of cases these are calomel electrodes or silver-silver-chloride electrodes. In the first case the reference system is a mercury in a saturated KCL solution, in the second case it is a silver wire also in a saturated (sometimes also in a 1molar) KCL solution. The exact design of the electrodes is not of consequence in this context.

It is important to know that the disposal of a mercury electrode must be carried out carefully.

It is also important to know the reference potentials of these electrodes in relation to the hydrogen potential. Typically, a data sheet is enclosed with the electrode. As reference values can be assumed:

- Calomel electrode +242mV against NHE
- Silver-silver-chloride electrode +207mV against NHE at 3molar KCL
- Silver-silver-chloride electrode +203mV against NHE at 1molar KCL

To calculate the correct setpoint, the value of the reference electrode must be subtracted from the setpoint.

For example, when measuring with an Ag/AgCl electrode with 3molar KCL, this results in a setpoint of $-1260\text{mV} - 207\text{mV} = -1467\text{ mV}$, which must be set on the instrument.

The potentiostat has a toggle switch on the left side to set the polarity. This is to be switched down. The potentiometer underneath has a 10 and a 100 division. If the black button is turned to the right, one complete turn corresponds to 100 parts of the outer scale and 1 part of the 10 scale. In numbers:

The potential range of the potentiometer is 2V. One complete turn of the black knob means 0.2V. The value of -1467mV therefore means 7.33 turns. The inner scale in the small window shows the number 7, the outer scale is at approx. 33.

Now you can switch the right toggle switch from Cell off to Cell on. With this the target potential is applied to the steel plate. The exact value can now be set by checking the left potential display.

Now the steel plate is under a cathodic protection current. This current can be a few mA, but it can also be 40 or 50mA. It does not have to be on a constant value but can change over time.

Depending on the regulations, the steel plate now remains under this cathodic load for 3 or 4 weeks.

After that:

- the potentiostat is switched from cell on to cell off. The sample is then currentless. This is the first mandatory procedure.
- the connecting cables at the electrodes are removed.
- the electrodes are removed from the lid and stored safely.
- the glass vessel is emptied and the sample can be separated from the glass cylinder for further examination.