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Paints and varnishes — Guidelines for the determination of anticorrosive properties of organic coatings by accelerated cyclic electrochemical technique

Peintures et vernis — Lignes directrices pour la détermination des propriétés anticorrosives de revêtements organiques par une technique électrochimique cyclique accélérée

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and vanishes*.

Introduction

This International Standard describes the determination of the anticorrosive properties of organic coatings by means of the accelerated cyclic electrochemical technique (ACET). The method is based on the so called AC/DC/AC procedure. This technique allows comparing the protective and anticorrosive properties of different coating systems on metal in short times and in a qualitative and quantitative way. ACET consists of the application of cycles of EIS (electrochemical impedance spectroscopy) measurements, cathodic polarizations and potential relaxation. Degradation of the coating system is accelerated by the cathodic polarization. EIS and potential relaxation monitor the change of the coating system induced by the cathodic polarization. The technique evaluates the permeability of the coating and properties which can be attributed to adhesion to the substrate.

Paints and varnishes — Guidelines for the determination of anticorrosive properties of organic coatings by accelerated cyclic electrochemical technique

1 Scope

This International Standard gives guidelines on how to perform accelerated cyclic electrochemical technique (ACET) with organic protective coatings on metals.

This International Standard specifies:

— the instrumental assembly;

— the execution of an ACET test and the considerations relative to the samples and electrochemical cell, test parameters and procedure.

This International Standard also provides guidelines for the presentation of experimental results and other type of information obtained.

Some typical examples are shown in an informative annex.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 16773-1, *Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens — Part 1: Terms and definitions*

ISO 16773-2:—[1)], *Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens — Part 2: Collection of data*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16773-1 and the following apply.

3.1
cathodic polarization
application of a potential U_{pol} more negative than the open-circuit potential U_{ocp} for a fixed period of polarization time t_{pol}

Note 1 to entry: This value is defined by the operator.

1) To be published. Revision of ISO 16773-2:2007.

3.2
relaxation time
t_{relax}
time between the cathodic polarization and the beginning of the electrochemical impedance spectroscopy (EIS) measurement

Note 1 to entry: This value is defined by the operator.

3.3
cathodic disbonding
failure of adhesion between a coating and a metallic surface that is directly attributable to cathodic reaction 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

[SOURCE: ISO 15711:2003, definition 3.1]

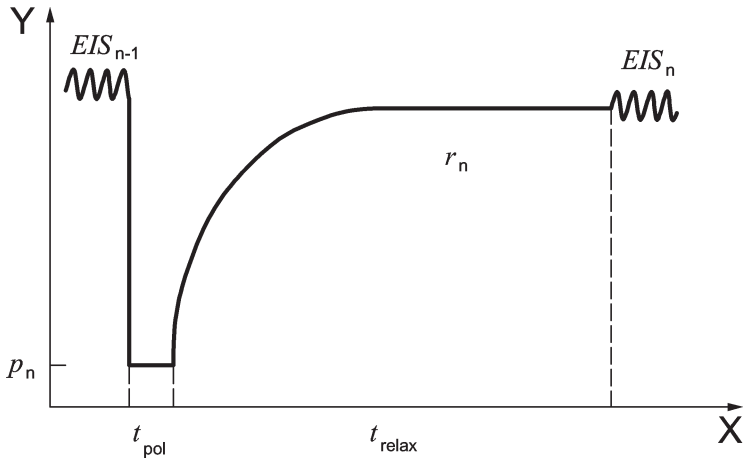
3.4
cathodic potential
P
potential difference between an applied potential U_{pol} and the open circuit potential U_{ocp}

4 Principle

4.1 General

The initial state of the coating system is characterized by an electrochemical impedance spectroscopy (EIS) measurement (EIS_{n-1} – see Figure 1). Then a cathodic polarization (P_n) is applied for a certain period of time (t_{pol}) followed by the relaxation process (r_n) over the time (t_{relax}). Finally the new state of the coating system is characterized by a second EIS measurement (EIS_n). These steps define a cycle which can be repeated n number of times (n is the number of cycles).

NOTE Typically six cycles are used.



Key
X time t, in min
Y potential U, in V

Figure 1 — ACET cycle

4.2 EIS measurement

A special type of potentiostat intended for EIS is connected to an electrochemical cell. A single-sinusoidal-waveform potential is applied and the resulting alternating current is measured. Both potential and current data are collected and analysed for amplitude and phase shift. More information about the procedure can be found in ISO 16773-2.

4.3 Cathodic polarization

A constant cathodic potential, U_{pol}, is applied. During the polarization, the electrolysis of water can take place if U_{pol} is negative enough:

$$H_2O\ (l) + e^- \rightarrow \tfrac{1}{2}H_2\ (g) + OH^-\ (aq)$$

The application of the cathodic potential will generate a stress on the protective coating.

If the electrolysis takes place at the interface of the coating to the metallic substrate, the hydroxide ions (OH^-) and/or hydrogen (H_2) can cause delamination between the metal and the coating.

4.4 Potential relaxation

The purpose of the relaxation process is to allow the formation of a new stable equilibrium before the next EIS measurement is performed. By recording the potential during the relaxation process, valuable additional information about the coating and the coating-metal interface can be obtained.

5 Apparatus

Use the electrochemical equipment specified in ISO 16773-2.

6 Specimens

6.1 Samples preparation

Proper preparation and preconditioning of coated specimens is critical for successful and reliable experimental data.

6.2 Environmental control

The coating should be applied and cured in accordance with the manufacturer's recommendation unless otherwise agreed upon between the parties involved. The film thickness should be as uniform as possible. The exact film thickness should be measured and reported (e.g. in accordance with ISO 2808). Temperature and humidity control during the application, curing, conditioning and measurement of organic coatings is crucial for a proper determination of the coating resistance.

For reliable measurements, temperature control should be equal to or better than ± 1 °C. For conditioning prior to measurement, an accuracy of ± 2 °C is sufficient for most cases. Each specimen should be kept under controlled conditions in order to prevent post-curing, degradation or any unintended irreversible modification of the coating.

The temperature of the specimens during measurements should be maintained constant to within ± 2 °C, preferably within ± 1 °C, at 23 °C, if not otherwise specified. Relative values for comparison between specimens outside these guidelines are acceptable if all the specimens are run under the same conditions. When the coating capacitance is the main parameter of interest, control of relative humidity during specimen conditioning is very important. To ensure accurate conditioning, the humidity should be (50 ± 5) % in accordance with ISO 3270, if not otherwise agreed.

6.3 Number of specimens and repeatability of results

Coatings are materials with certain inherent properties: holidays, inhomogeneous film thickness, and non-uniform distribution of pigments, fillers and other constituents. It is therefore necessary to test more than one panel. In most cases, a minimum of three replicate specimens is necessary for reliable results. It should be checked whether the uniformity between the different specimen plates is sufficient. It is quite common to find repeatability better than 10 % between the capacitance of replicate specimens, but it depends on the type of coating and the conditions of measurement. More replicates might be necessary to overcome uniformity problems.

Such checks should be the responsibility of the operator and should also be agreed between the parties involved.

Specimens undergoing a rapid change, caused by weathering or other effects producing degradation, can show a larger fluctuation and therefore a lower repeatability.

7 Procedure

7.1 EIS measurement

Perform the EIS measurement as specified in ISO 16773-2.

7.2 Cathodic polarization

Set a d.c. potential (see Figure 1) that allows the hydrolysis of the water at the pH of the electrolyte (approximately −1 V [SCE] at pH 7). Additionally, this potential should be fixed depending on the impedance of the coating; the larger it is the larger should be the magnitude of the applied cathodic potential. In general, potentials are applied in the range of −2 V to −4 V.

The cathodic polarization time varies depending on the quality of the coating. For high quality coatings with long term anticorrosive properties polarization time is typically between 20 min and 60 min.

7.3 Relaxation process

Disconnect the current supply and measure the relaxation potential over the time. The time is typically between 160 min and 180 min.

If the time of relaxation is too short, the equilibrium will not be reached and EIS measurement is not valid. If this time is too long, the system will evolve due to the migration of ions and the electrochemical reactions at the metallic substrate.

The study of the potential relaxation over time can provide useful information about the quality of the coating system, e.g. whether the cathodic reaction producing hydrogen (H_2) has taken place during the polarization. Hydrogen production can indicate a poor coating quality.

For interpretation of the plots, see Reference [3] and Reference [6].

7.4 Number of cycles

The cycle involving cathodic polarization/relaxation/EIS can be repeated either up to the degradation of the coating or it is possible to apply a given number of cycles. The number of cycles commonly used is 6. If no differences can be detected with 6 cycles, their number should be increased.

8 Data presentation

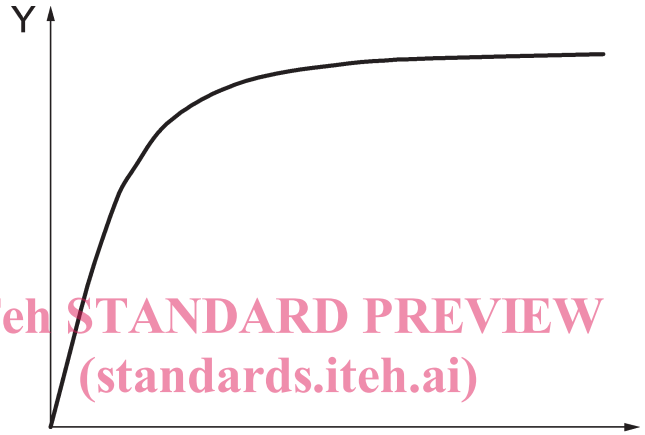
8.1 Graphics for EIS

Present the EIS measurements (Bode and Nyquist plots) as specified in ISO 16773-2:—, Clause 6. The number of cycles should be indicated in the graph if more than one impedance spectra are plotted together in one diagram.

Additionally specific values can be extracted from the EIS measurements and plotted against the number of cycles.

8.2 Graphics for the potential relaxation

Potential relaxation is plotted over time. A typical curve is shown in Figure 2.



Key

X relaxation time t_{relax}, in s

Y relaxation potential U_{relax}, in V

Figure 2 — Graph of relaxation potential

9 Precision

No precision data are currently available.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 17463);

c) the details on preparation of the test specimen, including

   1) the material (including thickness) and surface preparation of the substrate,

   2) the method of application of the tested coating to the substrate, including the duration and conditions of drying between layers in the case of a multi-coat system,

   3) the duration and conditions of drying (or stoving) of the coating material, and conditions of ageing, if applicable, before testing,