SLOVENSKI STANDARD
oSIST prEN ISO 17463:2021

01-junij-2021

Barve in laki - Smernice za ugotavljanje protikorozijskih lastnosti organskih premazov s pospešeno ciklično elektrokemijsko tehniko (ACET) (ISO/DIS 17463:2021)

Paints and varnishes - Guidelines for the determination of anticorrosive properties of organic coatings by accelerated cyclic electrochemical technique (ISO/DIS 17463:2021)

Beschichtungsstoffe - Leitfaden zur Bestimmung der antikorrosiven Eigenschaften organischer Beschichtungen durch beschleunigte zyklische elektrochemische Verfahren (ISO/DIS 17463:2021)

Peintures et vernis - Lignes directrices pour la détermination des propriétés anticorrosives de revêtements organiques par une technique électrochimique cyclique accélérée (ISO/DIS 17463:2021)

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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

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.

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 17463:2014), which has been technically revised. The main changes compared to the previous edition are as follows:

— The symbol for the potential has been changed from *U* to *E*;

— the specification of instrumental assembly has been deleted from the list in the scope;

— Bode plots and relaxation curves have been added as examples for the presentation of experimental results in the scope;

— the data presentation has been qualified to eventually Nyquist plots in 8.1;

— degradation has been stated more precisely to change in A.2 and A.3;

— the text has been editorially revised and the normative references have been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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

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

This document also provides guidelines for the presentation of experimental results such as Bode plots and relaxation curves and other type of information obtained.

Some typical examples are shown in Annex A.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 16773-2:2016, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
cathodic polarization
change of the electode potential in the negative direction caused by current flow

[SOURCE: ISO 8044:2020, 7.1.26]

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, 3.1]

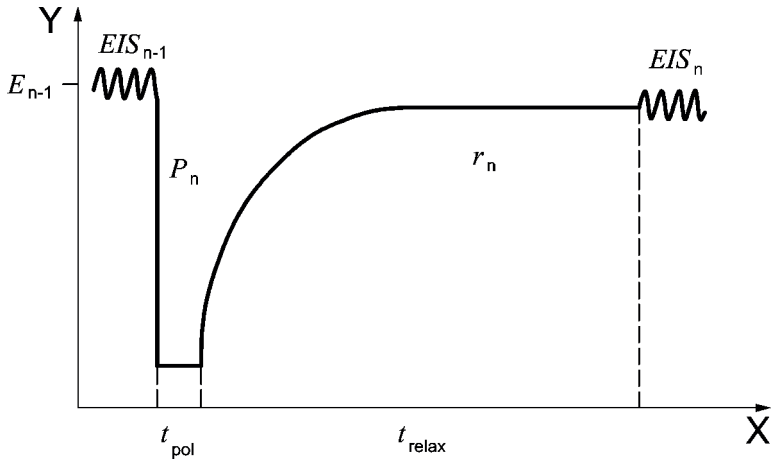
3.4
cathodic potential
E_P
potential applied negatively to the open circuit potential E_{ocp}

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
Y potential E

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, E_{pol}, is applied. During the polarization, the electrolysis of water can take place if E_{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.