SLOVENSKI STANDARD
SIST EN ISO 16773-2:2007

01-julij-2007

Barve in laki - Elektrokemijska impedanļna spektroskopija (EIS) vzorcev z visoko impedanco - 2. del: Zbiranje podatkov (ISO 16773-2:2007)

Paints and varnishes - Electrochemical impedance spectroscopy (EIS) on high-impedance coated specimens - Part 2: Collection of data (ISO 16773-2:2007)

Beschichtungsstoffe - Elektrochemische Impedanzspektroskopie (EIS) von beschichteten Proben mit hoher Impedanz - Teil 2: Datenerfassung (ISO 16773-2:2007)

Peintures et vernis - Spectroscopie d'impédance électrochimique (SIE) sur des éprouvettes revetues de haute impédance - Partie 2: Recueil des données (ISO 16773-2:2007)

Ta slovenski standard je istoveten z: **EN ISO 16773-2:2007**

ICS:

87.040	Barve in laki	Paints and varnishes

SIST EN ISO 16773-2:2007 **en;fr;de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 16773-2

April 2007

ICS 87.040

English Version

Paints and varnishes - Electrochemical impedance spectroscopy (EIS) on high-impedance coated specimens - Part 2: Collection of data (ISO 16773-2:2007)

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Foreword

This document (EN ISO 16773-2:2007) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 16773-2:2007 has been approved by CEN as EN ISO 16773-2:2007 without any modifications.

INTERNATIONAL STANDARD

ISO 16773-2

First edition
2007-04-01

Paints and varnishes — Electrochemical impedance spectroscopy (EIS) on high-impedance coated specimens —

Part 2: Collection of data

Peintures et vernis — Spectroscopie d'impédance électrochimique (SIE) sur des éprouvettes revêtues de haute impédance — Partie 2: Recueil des données

Reference number
ISO 16773-2:2007(E)

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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 16773-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 16773 consists of the following parts, under the general title *Paints and varnishes — Electrochemical impedance spectroscopy (EIS) on high-impedance coated specimens*:

— *Part 1: Terms and definitions*

— *Part 2: Collection of data*

— *Part 3: Processing and analysis of data from dummy cells* [1)]

— *Part 4: Examples of spectra of polymer-coated specimens* [1)]

1) In preparation.

Paints and varnishes — Electrochemical impedance spectroscopy (EIS) on high-impedance coated specimens —

Part 2: Collection of data

1 Scope

This part of ISO 16773 provides guidance on optimizing the collection of EIS data from high-impedance systems. High impedance in the context of intact coatings refers to systems with an impedance greater than $10^9\ \Omega{\cdot}cm^2$. This does not preclude measurements on systems with lower impedance.

This part of ISO 16773 deals with

— instrumental set-up: requirements and pit-falls;

— data validation: checking the measurement range and the accuracy of the data;

— performing an EIS measurement: specimen considerations and instrumental parameters;

— the experimental results: different methods of presenting EIS data.

Following the recommendations should ensure the acquisition of EIS data that can be used to study the performance of the specimen. It does not give guidelines for the interpretation of the data.

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

3 Terms and definitions

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

4 Principle

A so-called “confidence” test is described in order to check the suitability of the entire set-up and recommendations are given as to how to perform EIS experiments. For convenience, only potentiostatically controlled EIS measurements are described, although it is also possible to make EIS measurements under galvanostatic control.

A potentiostat is connected either to a dummy cell or to an electrochemical cell (with working, counter- and reference electrodes). A single-sinusoidal- or multi-sinusoidal-waveform potential, either in conjunction with a d.c. offset or not, is applied by the potentiostat to the dummy cell or to the electrochemical cell, and the resulting a.c. current is measured. Both potential and a.c. current data are collected and analysed for amplitude and phase shift. This can be done in a variety of ways, depending on the type of equipment used.

All data are presented and compared graphically, or computed for equivalent circuits. In the case of the dummy cell, the values of these equivalent components are compared to the actual cell components connected to the potentiostat and evaluated for coherence.

5 Electrochemical cell

5.1 General

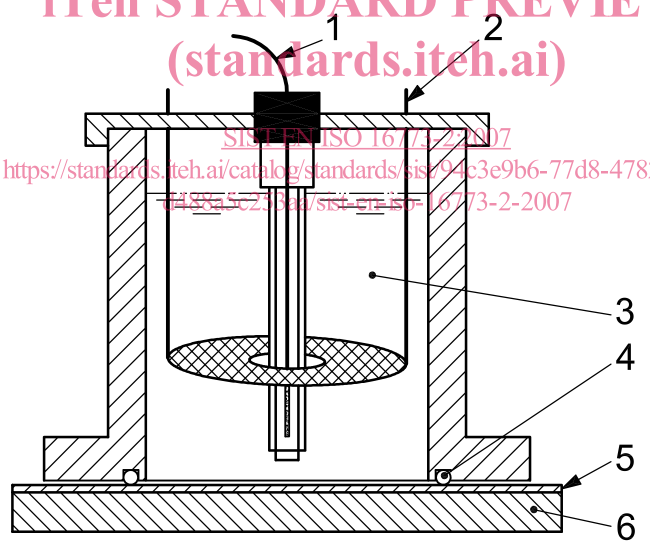
NOTE 1 Various types of measurement cell exist which are suitable for use with this part of ISO 16773. Most commonly used are two-electrode and three-electrode arrangements for measurements in an aqueous electrolyte.

The cell should be constructed of materials that will not corrode, otherwise deteriorate or contaminate the solution (e.g. PMMA, PTFE or glass). A material compatibility test should be carried out.

The cell should be leak-proof to ensure that the geometrical surface of the specimen does not change with time.

The cell should preferably be designed to allow the following items to be inserted into the electrolyte chamber: the working electrode, the reference electrode, the counter-electrode, a thermometer (for temperature control) and gas inlet/outlet tubes to modify the oxygen content of the electrolyte. When using an inert gas, a gas scrubber should be used.

An example of an electrochemical cell is shown in Figure 1.



Key

1 reference electrode
2 counter-electrode
3 electrolyte
4 O-ring
5 coating
6 working electrode

Figure 1 — Example of an electrochemical cell

NOTE 2 This drawing does not imply that other designs are unsuitable.

The components shown in Figure 1 are described in 5.2 to 5.4.

5.2 Electrodes

To perform EIS in aqueous solution, the more conventional set-up is composed of a three-electrode arrangement: a working electrode, a reference electrode and a counter-electrode.

Working electrode: A conductive substrate covered by the coating to be investigated. A large surface area is preferred to better take into account any defects and to decrease the impedance of the system to give a better signal-to-noise ratio.

Counter-electrode: Inert material such as platinum with a large surface area oriented parallel to the working electrode in order to ensure a homogeneous current distribution.

Reference electrode: A low-impedance and low-noise reference electrode is recommended [in the context of this part of ISO 16773, a salt bridge (e.g. Luggin capillary) is not required]. The potential of the reference electrode should be checked periodically to control the accuracy of the electrode and its stability with time. At very high frequencies, the presence of the reference electrode can induce some spurious effects.

NOTE 1 To improve the quality of the high-frequency signal, a platinum wire with a capacitor may be placed in parallel with the reference electrode. The capacitor ensures that the d.c. potential is coming from the reference electrode and a.c. potential from the platinum wire.

NOTE 2 For specific applications it can be acceptable to use a pseudo-reference electrode consisting of an inert material such as a high-nickel alloy or a chloridized silver wire. Pseudo-reference electrodes are useful for measurements in the field, where a reference electrode can be easily broken.

5.3 Exposed area

The exposed area should be accurately known, constant with time, and should be adequate for the investigation. Large areas make the measurement more sensitive for single defects (pores) and give better signal-to-noise ratio.

5.4 Electrolyte

The resistance of the solution should be low in comparison to the impedance of the investigated system. Different types of electrolyte can be used. Non-aggressive electrolytes can be employed to characterize the properties of the system without introducing corrosion. An aggressive solution may be selected to characterize the corrosion resistance of the system. The electrolyte should be chosen with the end use of the coating in mind.

6 Procedure

6.1 Grounding

An EIS instrument consists of a potentiostat, a computer and a module or instrument specifically required for the impedance measurement.

Electrical grounding considerations between the instruments, the specimen and the environment are important, both for the safety of the operator and the acquisition of as accurate and noise-free EIS data as possible.

a) The safety of the operator is important. The chassis of the EIS instrument should be connected to ground to avoid a potentially lethal electrical shock if the instrument malfunctions. The chassis is normally grounded through the connection to the a.c. mains. Under no circumstances should this connection to ground be bypassed.

b) In most cases, the coated specimen is tested in the laboratory in an electrochemical cell such as that described in 5.1 in which the specimen is electrically isolated from ground, or “floating”. This is the simplest case with no special consideration needed for connection of the instrument to the specimen.