BS EN ISO 16773-1:2016



BSI Standards Publication

Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens

Part 1: Terms and definitions



National foreword

This British Standard is the UK implementation of EN ISO 16773-1:2016. It supersedes BS EN ISO 16773-1:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 85633 4

ICS 87.040

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2016.

Amendments/corrigenda issued since publication

Date Text affected

EUROPEAN STANDARD NORME EUROPÉENNE

EN ISO 16773-1

EUROPÄISCHE NORM

April 2016

ICS 87.040

Supersedes EN ISO 16773-1:2007

English Version

Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens - Part 1: Terms and definitions (ISO 16773-1:2016)

Spectroscopie d'impédance électrochimique (SIE) sur des éprouvettes métalliques revêtues et non revêtues -Partie 1: Termes et définitions (ISO 16773-1:2016)

Elektrochemische Impedanzspektroskopie (EIS) an beschichteten und unbeschichteten metallischen Proben - Teil 1: Begriffe (ISO 16773-1:2016)

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European foreword

This document (EN ISO 16773-1:2016) 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of an identical text or by endorsement, at the latest by October 2016, and conflicting national standards shall be withdrawn at the latest by October 2016.

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The text of ISO 16773-1:2016 has been approved by CEN as EN ISO 16773-1:2016 without any modification.

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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.

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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 varnishes*.

This second edition cancels and replaces the first edition (ISO 16773-1:2007), which has been technically revised. The main changes are the following:

- a) the introductory element of the title, *Paints and varnishes*, has been omitted because the scope is broadened to include metals and alloys and the main element of the title has been changed to: *Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens*;
- b) a reference to ISO/TR 16208 has been added;
- c) the terms are listed in alphabetical order;
- d) the definition for working electrode has been implemented from ISO/TR 16208;
- e) the units and symbols have added where applicable.

ISO 16773 consists of the following parts, under the general title *Electrochemical impedance spectroscopy* (EIS) on coated and uncoated metallic specimens:

- Part 1: Terms and definitions
- Part 2: Collection of data
- Part 3: Processing and analysis of data from dummy cells
- Part 4: Examples of spectra of polymer-coated and uncoated specimens

Introduction

ISO 16773 describes the application of electrochemical impedance spectroscopy (EIS). Although this International Standard was originally developed for coatings and major sections are specific for coatings, the general guidelines can be used also for uncoated samples. For uncoated samples extra information can be found in ISO/TR 16208.

This part of ISO 16773 defines terms used in electrochemical impedance spectroscopy.

ISO 16773-2 describes an experimental procedure for testing laboratory instrumentation for collecting and presenting EIS data collected under potentiostatic control with a focus on high-impedance organic coatings on metal surfaces. ISO 16773-2 specifies a dummy cell that models the properties of a high-impedance system. It gives a test procedure and set-up parameters for the collection of impedance data from the dummy cell and the coated metal specimens. It outlines a procedure for comparing the recorded spectra with the theoretical data for the dummy cell in order to establish guidelines for acceptable instrumental accuracy and limitations. It does not provide any guidance on data interpretation.

ISO 16773-3 specifies the procedure used for the evaluation of the experimental results obtained from dummy cells which simulate high-impedance coated samples and it gives acceptance criteria for the values obtained.

ISO 16773-4, which is informative in nature, includes some background on impedance spectra of coated metal specimens and some typical examples of spectra from actual coatings.

ISO/TR 16208 describes basic principles of electrochemical impedance spectroscopy (EIS) specially focusing on the corrosion of metallic materials. It also deals with how to use electrochemical apparatus, set up and connect electrical instruments, present measured data, and analyse results.

Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens —

Part 1:

Terms and definitions

1 Scope

This part of ISO 16773 defines terms for electrochemical impedance spectroscopy (EIS) for use in the other parts of ISO 16773.

2 Terms and definitions

2.1

peak-to-peak amplitude

 $2U_0$

maximum value between maximum and minimum excitation of the applied a.c. perturbation signal

Note 1 to entry: The peak-to-peak amplitude is usually expressed in millivolts.

2.2

rms amplitude

 $U_{\rm rms}$

root-mean-square (i.e. effective) value of the applied a.c. perturbation signal

Note 1 to entry: This is the peak-to-peak value of the a.c. amplitude, divided by $2 \times \sqrt{2}$.

2.3

Bode plot

curves of (a) *phase angle* (2.36) versus the logarithm of the applied frequency and (b) the logarithm of the *magnitude of the impedance* (2.28) |Z| versus the logarithm of the applied frequency

2.4

charge transfer resistance

 R_{ct}

resistance of the resistor representing the metal-electrolyte interface characteristics in the *equivalent circuit* (2.18)

Note 1 to entry: The charge transfer resistance is usually expressed in ohms. The specific charge transfer resistance is usually expressed in ohms times square centimetres.

2.5

coating

layer formed from a single or multiple application of a coating material to a substrate

[SOURCE: ISO 4618:2014, 2.50.1]

2.6

coating

process of applying a coat

Note 1 to entry: The use of the term "coating" for "coating material" is deprecated.

[SOURCE: ISO 4618:2014, 2.50.2]

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2.7

coating capacitance

 C_{c}

capacitance of the capacitor representing the electrical capacitance of the *coating* (2.5) in the *equivalent circuit* (2.18)

Note 1 to entry: The coating capacitance is usually expressed in nanofarads. The specific coating capacitance is usually expressed in nanofarads per square centimetre.

Note 2 to entry: For coatings which do not have ideal capacitance, the constant phase element (CPE) is frequently used. The use of a constant phase element does not have a physical meaning.

28

coating resistance

 $R_{\rm c}$

resistance (2.40) of the resistor representing the electrical resistance of the coating (2.5) in the equivalent circuit (2.18)

Note 1 to entry: The coating resistance is usually expressed in gigaohms. The specific coating resistance is usually expressed in gigaohms times square centimetres.

2.9

corrosion potential

 $U_{\rm cor}$

potential of a corroding surface at which the rate of oxidation (corrosion) and the rate of reduction of the one or more oxidants are equal

Note 1 to entry: This is also known as the mixed potential or the rest potential.

Note 2 to entry: This potential is measured relative to a reference electrode (2.39) under open-circuit conditions.

Note 3 to entry: The corrosion potential is usually expressed in volts.

2.10

corrosion rate

amount of metal lost in unit time

Note 1 to entry: The corrosion rate is usually expressed in millimetres per year.

2.11

counter electrode

CE

inert electrode in the *electrochemical cell* (2.15) through which the current passes from or to the *working electrode* (2.46)

2.12

double-layer capacitance

 C_{d1}

capacitance of the capacitor representing the metal-electrolyte interface characteristics in the *equivalent circuit* (2.18)

Note 1 to entry: The double-layer capacitance is usually expressed in microfarads. The specific double-layer capacitance is usually expressed in microfarads per square centimetre.

2.13

dummy cell

printed-circuit board with mounted electrical components representing the *equivalent circuit* (2.18) and with connection points to the measuring instrument

2.14

electrical zero-reference

voltage with respect to which all voltages within the electrochemical equipment are referenced

2.15

electrochemical cell

system consisting of at least two electrodes in an electrolyte

2.16

electrochemical impedance spectroscopy

EIS

electrochemical technique which allows the impedance spectrum of an electrochemical system to be recorded as a function of the frequency of the applied signal, and the spectrum thus obtained to be analysed by transfer function analysis

2.17

electromagnetic noise

electrical noise in the current and/or voltage signal in a circuit whose origin is stray electromagnetic radiation from nearby electrical devices

2.18

equivalent circuit

electrical circuit modelling the *impedance* (2.23) of the specimen under test

Note 1 to entry: For example, this could be a network, consisting of elements such as a resistor, a capacitor and an inductor, which has the same impedance spectrum (i.e. the same response to a perturbation) as the electrochemical system.

2.19

error estimate

percentage deviation from zero of the recorded data, or the percentage deviation of the data from the absolute values of the elements of the dummy cell used

Note 1 to entry: The percentage deviation from zero of the recorded data is determined by subtracting the data from the theoretical equivalent-circuit data and dividing it by the latter. This method is only suitable for dummy cell measurements.

Note 2 to entry: The percentage deviation from the absolute values of the elements of the dummy cell used is determined by calculating the values of the equivalent-circuit elements from the recorded spectra. It is expressed as the percentage deviation from the absolute values of the elements of the dummy cell.

2.20

Faraday cage

metallic cabinet, used to reduce electromagnetic interference to the *electrochemical cell* (2.15) (or circuit), that completely encases the cell and is grounded to the instrument earth

2.21

galvanostat

electronic instrument which controls the current through the *working electrode* ($\underline{2.46}$) and *counter electrode* ($\underline{2.11}$) and which measures the resulting potential at the working electrode with respect to a *reference electrode* ($\underline{2.39}$)

2.22

ground

electronic reference point

Note 1 to entry: Voltage in the circuit is measured with respect to this reference point. In an electrochemical system, there could be several types of ground:

- a) Earth ground, which is a connection to earth. The "protective ground pin" in a.c. receptacles is connected to earth. The chassis of a desktop computer connected to the a.c. mains will be grounded in this way.
- b) Floating ground, which in an electrochemical system (instrumentation and cell) is one which is not actually connected to earth.
- c) Signal ground, which is the reference point in the electronic circuit of a *potentiostat* (2.38). A signal ground can be earthed or floating, depending upon the potentiostat design.

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d) Virtual ground, which is a point, generally the *working electrode* (2.46), maintained at a voltage equivalent to ground by an operational amplifier.

2.23

impedance

7

frequency-dependent, complex-number proportionality factor, $\Delta U/\Delta I$, between the applied alternating current voltage U (or current I) and the response current (or potential) in an *electrochemical cell* (2.15)

Note 1 to entry: This factor is the impedance only when the perturbation and response are linearly related (the value of the factor is independent of the magnitude of the perturbation) and the response is caused only by the perturbation. The value of the impedance can be related to the *corrosion rate* (2.10) when measurement is made at the *corrosion potential* (2.9).

Note 2 to entry: The impedance is usually expressed in ohms. The specific impedance is usually expressed in ohms times square centimetres.

2.24

IR drop

voltage drop in the electrolyte between the *working reference* (2.46) and the *reference electrode* (2.39), caused by the *resistance* (2.40) of the electrolyte and the distance between the electrodes

2.25

linear system

system in which the response to a perturbation is directly proportional to the perturbation

2.26

linear-system analysis

processing of the response of a *linear system* (2.25) to a perturbation

2.27

Lissajous figure

graphical representation of the response of an *electrochemical cell* (2.15) to a sine/cosine excitation, constructed by plotting the current against the voltage on mutually perpendicular axes

2.28

magnitude of the impedance modulus of the impedance

square root of the sum of the squares of the real and imaginary components of the *impedance* (2.23)

Note 1 to entry: It is given by $|Z| = [(z')^2 + (z'')^2]^{1/2}$

where

- *Z* is the complex impedance;
- z' is the real part of the impedance;
- z'' is the imaginary part of the impedance.

Note 2 to entry: The magnitude of impedance is usually expressed in ohms. The specific magnitude of impedance is usually expressed in ohms times square centimetres.

2.29

multi-sine analysis

application of a wave made up of several sine waves of different frequencies and measurement of the response at each frequency simultaneously

2.30

non-linear system

system in which the response to a perturbation is not directly proportional to the perturbation

2.31

non-linear system analysis

processing of the response of a *non-linear system* (2.30) to a perturbation

Note 1 to entry: If the amplitude of the applied perturbation is low enough, the system can be treated as a *linear system* (2.25).

2.32

Nyquist plot

negative of the imaginary component z'' of the *impedance* Z (2.23) plotted against the real component z' of the impedance

2.33

open-circuit potential

 $U_{\rm ocn}$

working electrode (2.46) potential, measured with respect to a reference electrode (2.39), when no current is flowing to or from the reference electrode

Note 1 to entry: The open-circuit potential is usually expressed in volts.

2.34

oxide laver

surface layer formed by the reaction of a metal with oxygen or oxygen compounds

2.35

phase

distance between the position of an amplitude crest of a wave train and a reference position

2.36

phase angle

Œ

phase (2.35) difference, expressed as an angle, between a voltage and current recurring periodically at the same frequency

Note 1 to entry: The phase angle is usually expressed in degrees.

2.37

polarization resistance

 $R_{\rm pol}$

slope, dU/dI, at the corrosion potential (2.9), of a potential, U, versus current, I, curve

Note 1 to entry: The polarization resistance is usually expressed in ohms. The specific polarization resistance is usually expressed in ohms times square centimetres.

2.38

potentiostat

electronic instrument for automatically maintaining the *working electrode* (2.46) in an electrolyte at a controlled potential with respect to a *reference electrode* (2.39) and for measuring the resulting current between the working electrode and the counter-electrode

2.39

reference electrode

RE

electrode having a stable and reproducible potential that is used as a reference in the measurement of electrode potentials

Note 1 to entry: This electrode has to have a potential which is thermodynamically stable with respect to that of the standard hydrogen electrode.

Note 2 to entry: Common reference electrodes are: saturated calomel electrode (SCE), silver/silver chloride electrode (Ag/AgCl), copper/copper sulfate electrode ($Cu/CuSO_4$).

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2.40

resistance

R

property of a circuit element by virtue of which it impedes the flow of electrical current through it

Note 1 to entry: The resistance is usually expressed in ohms.

2.41

salt bridge

laboratory device used to connect the *reference electrode* (2.39) to the *working electrode* (2.46) and to separate the reference electrode from the test solution (e.g. electrolyte)

Note 1 to entry: Commonly, a Luggin capillary is used.

2.42

shielding

blocking of *electromagnetic noise* (2.17) by encasing signal-carrying conductors or devices (e.g. a cell) in a conductive material which can be grounded or acted on by a signal equivalent to the signal of interest

2.43

single-sine analysis

application of a single sine wave of a given frequency and measurement of the response at that frequency

2.44

system-response analysis

analysing the response of a system to a perturbation

2.45

transfer-function analysis

technique whereby a property of the system under investigation is measured by perturbing the system by applying an a.c. input and analysing the *phase* (2.35) and amplitude of the response to determine the transfer function, i.e. *impedance* (2.23) of the system

2.46

working electrode

WE

test or specimen electrode in an electrochemical cell (2.15)

Bibliography

- [1] ISO 4618:2014, Paints and varnishes Terms and definitions
- [2] ISO/TR 16208, Corrosion of metals and alloys Test method for corrosion of materials by electrochemical impedance measurements





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