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

Part 1:
Terms and definitions

Peintures et vernis — Spectroscopie d'impédance électrochimique (SIE) sur des éprouvettes revêtues de haute impédance —

Partie 1: Termes et définitions

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Foreword

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ISO 16773-1 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]

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1) In preparation.

Introduction

ISO 16773 describes a procedure for the evaluation of protective anticorrosion coatings by using electrochemical impedance spectroscopy (EIS).

Part 1 defines terms used in electrochemical impedance spectroscopy.

Part 2 describes an experimental procedure for testing laboratory instrumentation for collecting and presenting EIS data collected under potentiostatic control for high-impedance organic coatings on metal surfaces. Part 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.

Part 3 deals with 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.

Part 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.

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

Part 1:
Terms and definitions

1 Scope

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

2 Terms and definitions

2.1
peak-to-peak amplitude
maximum value between maximum and minimum excitation of the applied a.c. perturbation signal

2.2
rms amplitude
root-mean-square (i.e. effective) value of the applied a.c. perturbation signal

NOTE 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 versus the logarithm of the applied frequency and (b) the logarithm of the magnitude of the impedance $|Z|$ versus the logarithm of the applied frequency

2.4
coating
continuous layer formed from a single or multiple application of a coating material to a substrate

[ISO 4618:2006]

2.5
corrosion potential
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 This is also known as the mixed potential or the rest potential.

NOTE 2 This potential is measured relative to a reference electrode under open-circuit conditions.

2.6
corrosion rate
amount of metal lost in unit time

NOTE This can be expressed, for example, as the change in mass per unit area per unit time.

2.7
counter-electrode
inert electrode in the electrochemical cell through which the current passes from or to the working electrode

2.8
coating capacitance
capacitance of the capacitor representing the electrical capacitance of the coating in the equivalent circuit

2.9
coating resistance
resistance of the resistor representing the electrical resistance of the coating in the equivalent circuit

2.10
double-layer capacitance
capacitance of the capacitor representing the metal-electrolyte interface characteristics in the equivalent circuit

2.11
charge transfer resistance
resistance of the resistor representing the metal-electrolyte interface characteristics in the equivalent circuit

2.12
dummy cell
printed-circuit board with mounted electrical components representing the equivalent circuit and with connection points to the measuring instrument

2.13
electrical zero-reference
voltage with respect to which all voltages within the electrochemical equipment are referenced

2.14
electromagnetic noise
electrical noise in the current and/or voltage signal in a circuit whose origin is stray electromagnetic radiation from nearby electrical devices

2.15
electrochemical cell
system consisting of at least two electrodes in an electrolyte

NOTE For the purposes of ISO 16773, the working electrode is coated to give it a very high impedance.

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
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 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 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.

2.18
equivalent circuit
electrical circuit modelling the impedance of the specimen under test

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

2.20
galvanostat
electronic instrument which controls the current through the working electrode and counter-electrode and which measures the resulting potential at the working electrode with respect to a reference electrode

2.21
ground
electronic reference point

NOTE 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. A signal ground can be earthed or floating, depending upon the potentiostat design.

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

2.22
***IR* drop**
voltage drop in the electrolyte between the working and the reference electrode, caused by the resistance of the electrolyte and the distance between the electrodes

2.23
impedance
frequency-dependent, complex-number proportionality factor, $\Delta U/\Delta I$, between the applied potential U (or current I) and the response current (or potential) in an electrochemical cell

NOTE 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 when measurement is made at the corrosion potential.

2.24
linear system
system in which the response to a perturbation is directly proportional to the perturbation

2.25
linear-system analysis
processing of the response of a linear system to a perturbation

2.26
Lissajou figure
graphical representation of the response of an electrochemical cell to a sine/cosine excitation, constructed by plotting the current against the voltage on mutually perpendicular axes

2.27
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

NOTE It is given by $|Z| = \left[(z')^2 + (z'')^2\right]^{1/2}$

where

Z is the complex impedance;

z' is the real part of the impedance;

z'' is the imaginary part of the impedance.

2.28
non-linear system
system in which the response to a perturbation is not directly proportional to the perturbation

2.29
non-linear system analysis
processing of the response of a non-linear system to a perturbation

NOTE If the amplitude of the applied perturbation is low enough, the system can be treated as a linear system.

2.30
Nyquist plot
negative of the imaginary component z'' of the impedance Z plotted against the real component z' of the impedance

2.31
open-circuit potential
working electrode potential, measured with respect to a reference electrode, when no current is flowing to or from the reference electrode

2.32
oxide layer
surface layer formed by the reaction of a metal with oxygen or oxygen compounds

2.33
phase angle
phase difference, expressed as an angle, between a voltage and current recurring periodically at the same frequency

2.34
phase
distance between the position of an amplitude crest of a wave train and a reference position

2.35
polarization resistance
slope, $\mathrm{d}U/\mathrm{d}I$, at the corrosion potential, of a potential, U, versus current, I, curve

2.36

potentiostat

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

2.37

reference electrode

electrode of reproducible potential to which an unknown electrode potential can be referenced

NOTE This electrode has to have a potential which is thermodynamically stable with respect to that of the standard hydrogen electrode.

2.38

resistance

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

2.39

shielding

blocking of electromagnetic noise 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

2.40

single-sine analysis

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

2.41

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

2.42

system-response analysis

analysing the response of a system to a perturbation

2.43

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 and amplitude of the response to determine the transfer function, i.e. impedance, of the system

2.44

working electrode

substrate of the coated specimen under investigation