
Paints and varnishes — Electrochemical measurement of the protection provided to steel by paint coatings — Current interrupter (CI) technique, relaxation voltammetry (RV) technique and DC transient (DCT) measurements

Peintures et vernis — Mesurage électrochimique de la protection apportée à l'acier par des revêtements de peinture — Technique du courant interrompu (CI), voltamétrie de relaxation (VR) et mesurages de courants continus transitoires (CCT)





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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

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Introduction

Quantitative assessment of protection performance of organic coatings has been required in industry, for example for evaluating the durability of organic coatings or judging the life of protective coatings. Electrochemical methods can be used for these purposes. The current interrupter (CI) technique, relaxation voltammetry (RV) and DC transient (DCT) measurements are simple techniques giving effective data which are comparable with electrochemical impedance spectroscopy (EIS) in principle.

An advantage is that the principle is simple and time for one measurement is short.

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1 Scope

This International Standard specifies the procedure for evaluation of the experimental set-up of electrochemical measurements on high-impedance coated samples using methods that are based on the current interrupter (CI) technique, relaxation voltammetry (RV) or DC transient (DCT) measurements.

It provides specific definitions and guidance on optimizing the collection of CI, RV and DCT data from high-impedance systems. High impedance in the context of intact coatings refers to systems with an impedance greater than $10^9 \Omega/\text{cm}^2$. This does not preclude measurements on systems with lower impedance. This International Standard deals in particular with:

- instrumental set-up: requirements and shortcomings;
- data validation: checking the measurement range and the accuracy of the data;
- performing CI, RV, DCT measurements: specimen considerations and instrumental parameters;
- the experimental results: different methods of presenting CI, RV and DCT data.

Following the recommendations should ensure the acquisition of CI, RV and DCT data that can be used to study the performance of the specimen. This International Standard 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 and the following apply.

3.1

excitation

application of a voltage, U_{exc} , or current, I_{exc} , to force the system into a new state

3.2

current interrupter method

CI method

electrochemical technique which allows the relaxation potential of an electrochemical system to be recorded as a function of time just after the excitation current is interrupted and the potential decay curve obtained to be analysed

3.3
relaxation voltammetry
RV
electrochemical technique in which a potential is applied for a given excitation time and then the current is interrupted and just the potential is monitored as a function of time

3.4
DC transient measurement
DCT measurement
electrochemical technique in which a coated sample is charged such that the potential is shifted and then the sample is isolated and the potential is monitored against time while the potential returns to the open-circuit potential

3.5
potential/time decay curve
curve of potential, $U(t)$, plotted over time, while potential is changing after a defined excitation is removed

3.6
sampling rate
number of data points per time interval

3.7
time constant
product of resistor and capacitor values used in equivalent circuits, which has the dimensions of time

4 Apparatus

4.1 Faraday cage

The Faraday cage shall be constructed of metal sheet which reduces electromagnetic interference to the electrochemical cell (or circuit), and shall be connected to the instrument earth.

4.2 Cell for sample, including electrode holder

NOTE 1 Various types of measurement cell exist which are suitable for use with this International Standard. 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 shall 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.

Sometimes a thermometer (for temperature control) and gas inlet/outlet tubes to modify the oxygen content of the electrolyte are used. When using an inert gas, a gas scrubber should be used.

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