

TECHNICAL REPORT
RAPPORT TECHNIQUE
TECHNISCHER REPORT

CEN ISO/TR 5602

November 2022

ICS 87.040

English Version

Sources of error in the use of electrochemical impedance spectroscopy for the investigation of coatings and other materials (ISO/TR 5602:2021)

Sources d'erreur dans l'utilisation de la spectroscopie d'impédance électrochimique pour l'étude des revêtements et autres matériaux (ISO/TR 5602:2021)

Fehlerquellen bei der Anwendung elektrochemischer Elektroimpedanzspektroskopie bei der Untersuchung von Beschichtungen und anderer Stoffe (ISO/TR 5602:2021)

This Technical Report was approved by CEN on 21 November 2022. It has been drawn up by the Technical Committee CEN/TC 139.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

The text of ISO/TR 5602:2021 has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TR 5602:2022 by Technical Committee CEN/TC 139 "Paints and varnishes" the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

Endorsement notice

The text of ISO/TR 5602:2021 has been approved by CEN as CEN ISO/TR 5602:2022 without any modification.

Contents

	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Error in the make-up of the measuring cell	1
4.1 Roughness of the surface	1
4.2 O-ring — Considerations about the precise determination of the exposed area	3
4.3 Faulty cell make-up	7
4.3.1 Optically detectable leaks	7
4.3.2 Optically non-detectable causes	7
4.4 Reference electrodes	9
4.4.1 General information on the distance between the reference and working electrodes	9
4.4.2 Shielding	11
4.4.3 Air bubble in the reference electrode	11
4.4.4 Poisoning of the reference electrode	11
4.4.5 Bleeding of the reference electrode	11
4.5 Counter electrodes	11
4.5.1 Relative sizes	11
4.5.2 Reactive counter electrodes	11
4.6 Gas inclusions in the measuring cell	11
5 Faults caused by electronics incl. shielding	12
5.1 Faraday cage	12
5.2 Extended cable (without active shielding)	15
5.3 Cable breaks	16
5.4 Contact resistances between metallic contacts and the working electrode/counter electrode	17
5.5 Inductivities	18
5.6 Measurement range switching	19
5.7 Scattering signals in power supply	20
5.8 Insufficient signal-to-noise ratio	22
5.9 Influence of peripheral devices	22
6 Parameter selection, measurement range limits	24
6.1 Open-lead test	24
6.2 Note on dummy cells – ISO 16773-3	24
6.3 Unsuitable amplitude	24
6.4 Insufficient frequency range	26
6.5 Repetition rate for subsequent measurements	27
7 Non-stationary measurement conditions	28
7.1 General	28
7.2 Temperature fluctuations	29
7.3 Electrolytic conductivity	31
7.4 Swelling	31
7.5 Drifting OCP	31
7.6 Corroding working electrode	33
7.7 Reactive counter electrodes	33
7.8 Gas formation at the counter electrode	33
8 Design and selection of equivalent circuit diagrams	34
8.1 Constant phase element	34
8.2 Multiple possibilities for the selection of equivalent circuits	35

8.3	Warburg impedance	37
9	Significance of measurement values from equivalent circuits.....	37
9.1	Measurement uncertainty.....	37
9.2	Plausibility analysis	38
10	Interpretation of the measurement values of various coating systems.....	39
10.1	Pre-treatment.....	39
10.2	Film thickness and measurement surface	40
10.3	Number of layers.....	41
10.4	Conditioning.....	45
10.5	Generic type of binder.....	45
11	Presentation of data.....	45
Annex A (informative) Calculation of the coating capacitance		48
Annex B (informative) Further information on the influence of the double-layer capacitance.....		49
Annex C (informative) Estimation of the order of magnitude of an apparent capacitance caused by corrosion.....		50
Bibliography.....		52

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*.

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

Electrochemical impedance spectroscopy is described in detail in ISO 16773-1 to ISO 16773-4. It became apparent during use of these standards that sources of error and measurement artefacts that lead to incorrect interpretations are not dealt with comprehensively. This document supplements the ISO 16773 series of standards to deal with this issue.

Sources of error in the use of electrochemical impedance spectroscopy for the investigation of coatings and other materials

1 Scope

This document describes the main sources of error in the use of electrochemical impedance spectroscopy for the investigation of coatings and other materials. The sources of error listed here include all process steps from the set-up of the sample with the measuring cell right through to evaluation.

NOTE The sources of error discussed here do not represent a complete list.

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 4618, *Paints and varnishes — Terms and definitions*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618, ISO 16773-1 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

limit impedance

minimum or maximum impedance that can be measured using the impedance spectrometer

3.2

limit frequency

minimum or maximum frequency that can be set on the impedance spectrometer

4 Error in the make-up of the measuring cell

4.1 Roughness of the surface

A wet and rough surface could conduct stray currents to a scratch or artificial defect, see [Figure 1](#). This could yield in a spectrum showing a much lower resistance than in reality. Examples of spectra are shown in [Figure 2](#).