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Corrosion of metals and alloys — Electrochemical measurements — Test method for monitoring atmospheric corrosion

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Contents

Page

Foreword ..... **v**
Introduction ..... **vi**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Summary of sensors **2**
5 Free corrosion current sensor **3**
5.1 Free corrosion current sensor description 3
5.2 Sensor geometry 3
5.3 Uniform corrosion current measurement 3
5.3.1 Use and conditions for uniform corrosion measurements 3
5.3.2 Method 1 — Sine wave excitation 3
5.3.3 Method 2 — Triangle wave excitation 3
5.3.4 Method 3 — Potential step excitation 3
5.4 Localized corrosion current measurement 4
5.5 Free corrosion rate and total free corrosion for sensors without coatings 4
5.5.1 Free corrosion current and current density 4
5.5.2 Free corrosion penetration rate 4
5.5.3 Free corrosion mass loss rate 5
5.5.4 Total free corrosion mass loss and corrosion penetration 5
5.6 Free corrosion current and total charge for sensors with coatings 5
5.6.1 Use and conditions for free corrosion measurements with coatings 5
5.6.2 Free corrosion current for a coated sensor 5
5.6.3 Free corrosion total charge for a coated sensor 5
5.7 Free corrosion sensor preparation 6
5.7.1 Considerations for free corrosion sensor surface preparation 6
5.7.2 Free corrosion sensors without coatings 6
5.7.3 Free corrosion sensors with coatings and surface treatments 6
5.8 Specification and inspection — Free corrosion sensors 7
5.8.1 Visual inspection 7
5.8.2 Sensor range and span 7
5.8.3 Electrical verification tests 7
5.8.4 Corrosion verification tests 7
6 Galvanic corrosion current sensor **7**
6.1 Galvanic corrosion current sensor description 7
6.2 Sensor geometry 8
6.3 Galvanic corrosion current measurements 8
6.3.1 Methods for galvanic corrosion current measurement 8
6.3.2 Method 1 — Zero-resistance ammeter 8
6.3.3 Method 2 — Precision resistor 8
6.4 Galvanic corrosion rate and total galvanic corrosion without coatings 8
6.4.1 Galvanic corrosion current 8
6.4.2 Galvanic corrosion rate for mass loss and corrosion penetration 8
6.4.3 Total galvanic corrosion mass loss and corrosion penetration 9
6.5 Galvanic corrosion rate and total galvanic corrosion with coatings 9
6.5.1 Use and conditions for galvanic corrosion measurements with coatings 9
6.5.2 Galvanic mass loss corrosion rate for a coated sensor 9
6.5.3 Total galvanic mass loss for a coated sensor 9
6.6 Galvanic corrosion sensor preparation 9
6.6.1 Considerations for galvanic corrosion sensor preparation 9
6.6.2 Galvanic corrosion sensors without coatings 9
6.6.3 Galvanic corrosion sensors with coatings and surface treatments 9

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https://standards.iteh.ai/catalog/standards/sist/a52a1e22a-f6f6-4f4b-955c-238c4d6d6d6e/iso-fdis-22858

6.7 Specification and inspection — Galvanic corrosion sensors 9
6.7.1 Visual, span and range inspection 9
6.7.2 Electrical verification tests 10
6.7.3 Corrosion verification tests 10

7 Thin film conductance sensors 10
7.1 Conductance sensor description 10
7.2 Sensor geometry 10
7.3 Surface conductance measurement method 10
7.4 Surface conductance sensor preparation 10
7.5 Specification and inspection — Conductance sensor 11
7.5.1 Visual, span and range inspection 11
7.5.2 Electrical verification tests 11
7.5.3 Conductive solution verification tests 11

8 Coating barrier property sensors 11
8.1 Coating barrier property sensor description 11
8.2 Coating barrier property measurements 11
8.3 Coating barrier property sensor preparation 12
8.3.1 Sensor preparation for coating 12
8.3.2 Coating test condition 12
8.4 Specification and inspection — Coating barrier property sensor 12
8.4.1 Visual, span and range inspection 12
8.4.2 Electrical measurements 12
8.4.3 Sensing system impedance verification tests 12

9 Atmospheric testing with electrochemical sensors 12
9.1 Types of atmospheric tests 12
9.2 Test arrangement 12
9.3 Test duration 12
9.4 Sensor selection 13
9.5 Sampling time interval 13
9.6 Date and time information 13

10 Test report 13
10.1 Test report guidance 13
10.2 Sensor information 13
10.3 Surface preparation 13
10.4 Test description 13
10.5 Sensor inspection 13
10.6 Data storage 14

Annex A (informative) **Example images of electrochemical sensors 15**

Annex B (informative) **Equivalent circuit analysis for two-electrode measurements 17**

Annex C (informative) **Example of reporting information 19**

Bibliography 21

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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.

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).

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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 156, *Corrosion of metals and alloys*.

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.

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Introduction

The purpose of this document is to provide instructions on the use of electrochemical sensors for monitoring atmospheric corrosion. These sensors are used to measure thin film electrolyte conductance, corrosion current or coating condition over long periods. This method permits the instantaneous evaluation of corrosion current that can be related to specific environmental conditions in real time. The instantaneous corrosion current measurements are not accessible using electrical resistance sensors or mass loss techniques. The technology described in this document complements other standard techniques for assessing atmospheric corrosion such as mass loss coupons, electrical resistance sensors or coated test panels (see ISO 8407 and ISO 4628-8). These continuous records of material condition can be useful for studying atmospheric corrosion, evaluating materials or managing assets[21][22][23][24][25][26][27][28][29].

This document was developed based on ANSI/NACE TM0416-2016.

This document is relevant to alloy and coating manufacturers and users in transportation, chemical process, energy and infrastructure applications.

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Corrosion of metals and alloys — Electrochemical measurements — Test method for monitoring atmospheric corrosion

1 Scope

This document specifies a test method for atmospheric corrosion measurements, using two-electrode electrochemical sensors.

It is applicable to measurements of the corrosion rate of uncoupled metal surfaces (i.e. "free" corrosion rate), galvanic corrosion rate, conductance of thin film solutions and barrier properties of organic coatings. It specifies electrochemical sensors that are used with or without organic coatings. The sensors are applicable to corrosion measurements made in laboratory test chambers, outdoor exposure sites and service environments.

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 4628 (all parts), *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance*

ISO 8044, *Corrosion of metals and alloys — Vocabulary*

ISO 9223, *Corrosion of metals and alloys — Corrosivity of atmospheres — Classification, determination and estimation*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618, ISO 4628 (all parts), ISO 8044, ISO 9223 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
electrical resistance sensor
device for measuring corrosion involving measurement of the ratio of the potential difference along a conductor and the current through the conductor

Note 1 to entry: ISO 15091:2019, 3.1, defines "electrical resistance" as the "ratio of the potential difference along a conductor and the current through the conductor".

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3.2
electrochemical sensor
device for measuring corrosion involving anodic and cathodic reactions

Note 1 to entry: ISO 8044:2020, 4.1, defines "electrochemical corrosion" as "corrosion involving at least one anodic reaction and one cathodic reaction".

3.3
electrode digit
single finger of an *interdigitated electrode* (3.5)

3.4
corrosion penetration
distance between the corroded surface of a metal and the original surface of the metal

Note 1 to entry: ISO 8044:2020, 3.11, defines "corrosion depth" as the "distance between a point on the surface of a metal affected by corrosion and the original surface of the metal".

3.5
interdigitated electrode
electronic conductors interlocked like fingers

3.6
sensor range
upper and lower measurement values

3.7
sensor span
difference between maximum and minimum measurement values

3.8
solution resistance
ratio of electrode potential increment to the corresponding current increment dependent on solution

3.9
thin film conductance
solution layer current transport capacity

Note 1 to entry: ISO 15091:2019, 3.3, defines "conductance" as the "reciprocal of the resistance".

3.10
zero-resistance ammeter
instrument used for current measurement between two electrodes with no potential drop between them

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4 Summary of sensors

The atmospheric corrosion measurements are made using three types of sensors to measure: a) free corrosion current, b) galvanic corrosion current and c) surface conductance.

Electrochemical sensors for atmospheric corrosion shall have a planar gage area. They are composed of two metallic electrodes separated by a dielectric material that electrically isolates the electrodes (see Figures A.1 and A.2). The electrochemical sensors have interdigitated electrode geometries and may be produced using composite laminate or thin film processes (see Annex A). The sensor gage area is defined as the area of the electrodes exposed to the environment.

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5 Free corrosion current sensor

5.1 Free corrosion current sensor description

Free corrosion rate measurements are obtained using two-electrode sensors that may have a variety of alloys, geometries and excitation techniques[22][23][24][25][27]. Two-electrode sensors for the measurement of free corrosion rate may be made from any alloy of interest. Both electrodes of the free corrosion current sensor shall be constructed of the same alloy. The electrical excitation of sensors shall be done by applying a voltage between the two electrodes. Voltage may be applied using a potentiostat or other electronic device designed to apply a controlled potential. During measurements, the voltage between the two electrodes is controlled and the current response recorded. Between measurements, the two electrodes of the sensor may be electrically shorted. The corrosion current measurement range and span will be dependent on the expected corrosion rates of the given sensor alloy in the environment.

5.2 Sensor geometry

The separation distance between the electrodes should be no more than 300 µm (see Figures A.1 and A.2). A high electrode digit length to width ratio minimizes the contribution of edge effects that distort current and potential distributions, and small width electrodes support a more uniform active measurement area under varying environmental conditions. An example length to width ratio is 10 and an example electrode digit width is 2 mm. Geometry and sensing areas for each electrode should be the same.

5.3 Uniform corrosion current measurement

5.3.1 Use and conditions for uniform corrosion measurements

For alloys that undergo uniform corrosion, such as a low alloy steel, polarization resistance may be obtained and free corrosion rate estimated by means of the Stern-Geary equation (see ISO 17475, ISO/TR 16208 and ASTM G59-97. For a simple equivalent circuit model of a two-electrode sensor, the polarization resistance may be approximated as half of the real impedance at a low frequency (see Annex B). This assumes that the solution resistance is small relative to the polarization resistance. This assumption may not be valid at low levels of corrosive contaminants or for very thin or discontinuous solution layers. Uniform corrosion measurements should be validated with mass loss or other coupon tests for environments classified as low corrosivity outdoor (C1) or medium corrosivity indoor (IC 3) or less (see ISO 9223, ISO 9224, ISO 9226 and ISO 11844-1). High solution resistance could result in an underestimation of corrosion rate. Polarization resistance shall be determined using the methods given in 5.3.2 to 5.3.4.

5.3.2 Method 1 — Sine wave excitation

The current response may be measured using a voltage sine wave excitation. The amplitude should be less than 30 mV. The excitation frequency shall be low enough, typically from 0,01 Hz to 10 Hz, to obtain a reasonable estimate of the polarization resistance. This method may require verification that the selected frequency yields, or correlates to, polarization resistances obtained using a full electrochemical impedance scan (see ISO/TR 16208 and ASTM G102-89).

5.3.3 Method 2 — Triangle wave excitation

The current response may be measured using a triangle wave voltage excitation with an amplitude not greater than 30 mV. The excitation signal shall have a ramp rate from 0,05 mV/sec to 10 mV/sec. This method may require verification that the selected waveform produces polarization resistances that correlate to those obtained using potentiodynamic scan methods (see ISO 17475 and ASTM G59-97).

5.3.4 Method 3 — Potential step excitation

The current response may be measured using potential steps and holds. A sufficient number of steps shall be used to obtain a linear fit to the voltage versus current response data over a potential range

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no greater than ±30 mV[22]. For each step, the hold time should be sufficient to obtain a steady-state current measurement. For each step, the current shall be measured after the hold time and be an average of multiple readings. This method may require verification that the selected excitation produces polarization resistances that correlate to polarization resistances obtained using potentiodynamic scan methods (see ASTM G59-97).

5.4 Localized corrosion current measurement

For alloys that corrode by localized mechanisms, such as aluminium alloy pitting, the impedance should be measured for a given sine wave voltage excitation, and the amplitude should be less than 30 mV. The impedance may be either the real component or modulus. The excitation frequency shall be within the range of 0,01 Hz to 10 Hz. In the case of localized corrosion processes, a constant of proportionality that empirically relates the measured impedance to the corrosion current is needed to make absolute estimates of corrosion rate.

5.5 Free corrosion rate and total free corrosion for sensors without coatings

5.5.1 Free corrosion current and current density

Free corrosion current density shall be reported as microampere per square centimetre (μA/cm^2). Current density shall be calculated using the free corrosion current and the area of one electrode. If one electrode is smaller, then the smallest electrode area shall be used. See Formula (1).

$$i_{\mathrm{corr}} = \frac{I_{\mathrm{corr}}}{A} \tag{1}$$

where

i_{corr} is the free corrosion current density, expressed as microampere per square centimetre (μA/cm^2);

I_{corr} is the free corrosion current, expressed as microampere (μA);

A is the electrode area, expressed as square centimetre (cm^2).

5.5.2 Free corrosion penetration rate

The free corrosion rate may also be converted to free corrosion penetration rate, but this shall only be done for alloys with uniform corrosion and shall not be used for alloys with localized corrosion such as pitting or intergranular corrosion. See Formula (2):

$$r_{\mathrm{t}} = K_1 \cdot \frac{i_{\mathrm{corr}}}{\rho} \cdot W_{\mathrm{e}} \tag{2}$$

where

r_{t} is the corrosion penetration rate of a metal, expressed as micrometre per year (μm/a);

K_1 is a constant of proportionality equal to 3,27, expressed as micrometre gram per microampere centimetre year ((μm · g)/(μA · cm · a)) (see ASTM G102-89);

ρ is the density of the metal, expressed as kilogram per cubic metre (g/cm^3);

W_{e} is the atomic weight of the metal divided by the valence of the oxidized metal atom, this is used as a dimensionless quantity (see ASTM G102-89).

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Methods are available for obtaining corrosion mass loss and penetration rate for alloys that use alloy equivalent weight and densities, but these methods are outside the scope of this document (see ASTM G102-89).

5.5.3 Free corrosion mass loss rate

Free corrosion current density measurement may be converted to mass loss corrosion rate. See Formula (3):

$$r_{\text{corr}} = K_2 \cdot i_{\text{corr}} \cdot W_{\text{e}} \tag{3}$$

where

r_{corr} is the corrosion mass loss rate of metal, expressed in gram per square metre per year ($g/(m^2 \cdot a)$);

K_2 is a the constant of proportionality equal to 3,268, expressed as gram square centimetre per microampere square metre year ($(g \cdot cm^2)/(\mu A \cdot m^2 \cdot a)$).

5.5.4 Total free corrosion mass loss and corrosion penetration

Time-based measurements of free corrosion current or corrosion rate may be integrated to obtain estimates of total charge passed or total mass loss, respectively. Total charge or mass loss shall be expressed in coulombs per square metre (C/m^2) or grams per square metre (g/m^2), respectively. Measures of thickness loss may also be reported as micrometres (µm), but shall only be reported for alloys with uniform corrosion. Total mass loss obtained from the sensor should be compared to the mass loss of specimens produced from the same alloy as the sensor (see ISO 8407).

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5.6 Free corrosion current and total charge for sensors with coatings

5.6.1 Use and conditions for free corrosion measurements with coatings

For use with coatings, the sensor should be mounted to form a planar surface that is greater than the sensor gage area along each edge (see Figures A.1 and A.2). This may be achieved by casting, potting or mounting the sensors.

The sensor responses are dependent on coating properties and coating defect area. The sensor response may change during the test as the coating degrades. Therefore, the sensor response is not a measure of uniform conditions over the complete gage area of the sensor.

5.6.2 Free corrosion current for a coated sensor

For a coated free corrosion sensor, the current should be measured for a given sine wave voltage excitation and the amplitude should be less than 30 mV. The excitation frequency shall be within the range of 0,01 Hz to 10 Hz.

Current shall be expressed as microamps (µA).

5.6.3 Free corrosion total charge for a coated sensor

Time-based measurements of current may be integrated to obtain estimates of total charge passed. Total charge shall be expressed as coulombs (C).

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