

Test Method for Monitoring Atmospheric Corrosion Rate by Electrochemical Measurements

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ABSTRACT

This standard test method provides guidance on the specification, selection, and use of sensors for monitoring atmospheric corrosion that are based on electrochemical techniques. The sensors may be used like more traditional mass loss coupons or painted test panels, but these sensors provide continuous records of contaminants, corrosion rates, or coating condition over time as opposed to singular cumulative measurements of mass loss or coating degradation. This method permits instantaneous evaluation of corrosion rates so that situations where changes in environmental conditions cause changes in the corrosion rate can be detected in real time. It addresses the use of electrochemical sensors in a bare metal condition or with protective coatings. It encompasses sensor elements for measurement of free corrosion, galvanic corrosion, and conductance for assessing atmospheric corrosion.

KEYWORDS

Atmospheric corrosion, galvanic, electrochemical, corrosion rate, conductance, sensor, TG 530.



Foreword

In NACE standards, the terms shall, must, should, and may are used in accordance with the definitions of these terms in the NACE Publications Style Manual, 4th ed., Paragraph 7.4.1.9. Shall and must are used to state mandatory requirements. The term should is used to state something considered good and is recommended but is not mandatory. The term may is used to state something considered optional.

The purpose of this standard test method is to provide guidance on the specification, selection, and use of sensors for monitoring atmospheric corrosion that are based on electrochemical techniques. The sensors may be used like more traditional mass loss coupons or painted test panels, but these sensors provide continuous records of contaminants, corrosion rates, or coating condition over time as opposed to singular cumulative measurements of mass loss or coating degradation. This method permits instantaneous evaluation of corrosion rates so that situations where changes in environmental conditions cause changes in the corrosion rate can be detected in real time. This is considered a substantive benefit as compared with mass loss methods. These continuous records of material condition are broadly applicable to studying atmospheric corrosion, evaluating materials, or managing assets. The method addresses the use of electrochemical sensors in a bare metal condition or with protective coatings. It encompasses sensor elements for measurement of free corrosion, galvanic corrosion, and conductance for assessing atmospheric corrosion. Atmospheric corrosivity measurements, using electrochemical-based sensors, provide a means to obtain instantaneous corrosion rate, surface contaminant, and coating property estimates over long exposure periods. The electrochemical-based sensors may be used to estimate free corrosion rate of specific alloys, galvanic corrosion rate of coupled materials, conductance of surface moisture layers, or the barrier properties of coatings. These sensors may be included in instrumentation used in accelerated test chambers, outdoor exposure sites, or service environments.

This standard test method for measuring atmospheric corrosion may be useful for material selection, establishing maintenance requirements, characterization of test chambers, site surveys, environmental severity mapping, asset monitoring, and investigating interactions between the environment, materials, and protection systems. These sensors and measurement techniques enable atmospheric corrosion monitoring systems for better design and management of mobile and fixed assets and infrastructure. These corrosion rate measurements may be used to characterize temporal and spatial variations of corrosivity throughout a given structure or for surveying different geographic locations.

The test method has the advantage of producing direct measures that are dependent on electrochemical reaction rates, conductance of thin film electrolytes, and coating performance over long periods, under atmospheric conditions. These instantaneous corrosion rate measurements are not readily accessible using electrical resistance and microbalance mass loss techniques. The technology described in the test method compliments other standard techniques for assessing atmospheric corrosion such as mass loss coupons, electrical resistance, and microbalance methods.¹⁻³ The electrochemical sensor techniques, described in this test method, have the benefit of providing continuous measures of instantaneous performance properties.

This NACE standard test method was prepared in 2016 by Task Group (TG) 530, "Test Method for Monitoring Atmospheric Corrosion Rate by Electrochemical Measurements." TG 530 is administered by Specific Technology Group (STG) 62, Corrosion Monitoring and Measurement—Science and Engineering Applications," and sponsored by STG 40, "Military and Aerospace Systems and Facilities," and STG 41, "Electric Utility Generation, Transmission, and Distribution." This standard is issued by NACE under the auspices of STG 62.

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Section 1: General

- 1.1 Scope:** This test method for making atmospheric corrosion measurements encompasses: 1) two-electrode electrochemical impedance, 2) zero resistance ammeter (ZRA), and 3) precision resistor current measurement techniques. The measurements can be used to quantify free corrosion, galvanic corrosion, conductance of surface contaminants (electrolyte layers), and properties of protective coatings. Factors important in specifying and selecting sensing elements and electrochemical measurement methods are provided. Sensing element types are described, along with excitation methods and data collection considerations. Methods are given to convert electrical responses to corrosion rate and cumulative corrosion measurements. Guidance is given for quality assurance practices, sensor preparation, and atmospheric corrosion testing. The test method also provides instruction for the preparation and testing of coatings using electrochemical sensors. Use and limitations of this method for determining the performance of materials and predicting corrosion behavior are described. Guidance is given for reporting sensor information, results, and environmental test parameters.
- 1.2 Limitations for Technical Use:** The electrochemical measurements can be used for comparative assessment of environments, alloys, and coatings. These sensors provide electrochemical measurements for estimating corrosion rate, but correlation and calibration may be required to establish absolute measures of corrosion for given environments and corrosion mechanisms. Caution should be exercised when making claims about expected performance for environments other than those tested, or when extrapolating data to predict service life based on accelerated or short duration performance testing. Electrochemical measurements used to estimate area averaged corrosion rates do not inherently provide a means to quantify localized corrosion damage and depth of penetration associated with localized corrosion mechanisms such as pitting and intergranular corrosion. Performance of electrochemical sensors and measurement systems will be dependent on the specific sensing elements, preparation process, and supporting electronics. Electrical measurements are described in this test method to establish measurement system specifications. The sensors covered by this standard have a wide range of uses, and therefore the method is not intended to be highly prescriptive for a given sensor technology or application. Parameters identified, but not specified should be considered by user and supplier in the design and use of these sensors for a given application. Prescriptive descriptions for verifying and validating measurement system performance for particular environments, sensor alloys, electrode geometries, and applications are beyond the scope of this test method. Comparative performance determinations should not be made using sensors with different geometries, excitation and acquisition hardware and software, or from different manufactures, unless comparable system responses have been validated. Use of electrochemical sensors to evaluate conductive coatings, such as electroplate, hot dip coatings, metalized films, or metal rich paints is outside the scope of this test method. Specific safety, durability, and regulatory requirements for the use and operation of the systems described in this document for any given application are outside the scope of the test method.
- 1.3 Applicability:** The electrochemical sensors are useful for comparative testing of materials in atmospheric corrosion tests including accelerated laboratory, outdoor exposure sites, and service environments. The electrochemical sensors provide continuous measures indicative of current environmental and material conditions. The test method can be used to assess the relative severity of corrosive environments using standard materials, or to assess material performance in specific environments. The electrochemical sensors can be used to quantify and track corrosion rate and estimate cumulative corrosion. The protective properties of coatings on metals and alloys can be continuously measured over long periods using the electrochemical sensors.

Section 2: Summary of Sensors

- 2.1 The atmospheric corrosion measurements included in this test method are: 1) two-electrode electrochemical impedance, 2) ZRA, and 3) precision resistor current measurement techniques. These measurement techniques and associated sensing elements can be used to measure free corrosion, galvanic corrosion, surface conductance, and coating properties (Table 1).
- 2.2 Atmospheric corrosion sensors have a nominally planar geometry and are composed of two electrodes separated by dielectric materials that protect and electrically isolate the electrode surfaces other than the sensing areas (Appendix A [nonmandatory], Figures A1 and A2). The two electrodes have interleaved geometries with a defined separation distance. These interleaved electrodes are referred to as interdigitated electrodes and they can be produced using laminates or thin films. Two-electrode composite laminate sensors can be fabricated using potted alloy materials to form interdigitated electrodes (IDE). Thin film or foil electrode sensors can also be produced by depositing or adhesively bonding metals and alloys onto dielectric substrates such as polymers, ceramics, or fiber reinforced polymers. Materials of construction are selected to be chemically resistant and durable in the exposure environment. The sensor gage area is defined as the area of the electrodes exposed to the environment.
- 2.3 The electrochemical sensors can be used individually or in combination to evaluate coatings in accelerated tests, outdoor exposures, or service environments (Figure A3). These measurement techniques and associated sensing elements can be used to measure the capability of coatings to control free corrosion, galvanic corrosion, and maintain barrier properties.

Table 1
Sensor Uses and Types of Measurement Techniques

Electrochemical Sensors	Measurement Method		
	Two-electrode impedance	Zero resistance ammeter	Precision resistor
Free corrosion	Yes	No	No
Galvanic corrosion	No	Yes	Yes
Surface conductance	Yes	No	No

Section 3: Free Corrosion Sensors

- 3.1 Free corrosion measurements are obtained using two-electrode sensors that may have a range of geometries and excitation techniques.⁴⁻⁶
- 3.2 Two-electrode sensors for measurement of free corrosion can be fabricated from any alloy of interest for a given application, or a reference alloy for determining the relative severity of a given environment. Both electrodes of the two-electrode free corrosion sensor shall be constructed of the same alloy.
- 3.3 The separation distance between the electrodes should be uniform and no more than 300 μm . A high electrode digit length to width ratio may minimize contribution of edge effects that distort current and potential distributions, and small width electrodes support 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.

- 3.4** Electrical excitation of sensors shall be done by applying a voltage signal between the two electrodes. During measurements, the relative potential between the two electrodes is controlled and the current response recorded.
- 3.5** Current range will depend on the alloy, electrode test area, amplitude of excitation voltage, and test environment. Spans for current measurements should be selected based on these factors. The corrosion rate measurement range (maximum and minimum measurements) and span (difference in maximum and minimum full-scale reading) will be dependent on the expected corrosion rates of the given sensor alloy.
- 3.6** Between measurements, the two electrodes of the sensor should be electrically shorted.
- 3.7** For alloys that undergo uniform corrosion, polarization resistance may be obtained and i_{corr} estimated by means of the Stern-Geary equation.⁷ For a simple equivalent circuit model of a two electrode sensor, the polarization resistance can be approximated as half of the real impedance (Appendix B [nonmandatory]). 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 electrolyte layers. High solution resistance results in an underestimation of corrosion rate. Polarization resistance shall be determined using the following methods.
- 3.7.1 Method 1 – Sine Wave Excitation.** Current response may be measured using a voltage sine wave excitation, and 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. The average of multiple full cycles should be used to obtain a current measurement. This method may require verification that the selected frequency yields, or correlates to polarization resistances obtained using a full electrochemical impedance scan.⁸
- 3.7.2 Method 2 – Triangle Wave Excitation.** 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 potential dynamic scan methods.⁷
- 3.7.3 Method 3 – Potential Step Excitation.** 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 no greater than ± 30 mV.⁵ 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 potential dynamic scan methods.⁷
- 3.8** For alloys that corrode by localized mechanisms such as 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 0.01–10 Hz. In the case of localized corrosion processes, a constant of proportionality that relates the measured impedance to the corrosion current is needed to make absolute estimates of corrosion rate.
- 3.9** Corrosion current (i_{corr}) shall be reported as mA/cm². Current density shall be measured using the area of one electrode. If one electrode is smaller, then the

smallest electrode area shall be used.

3.10 Free corrosion measurements may be converted to mass loss rate using Faraday's Law.⁹ Mass loss rate shall be expressed as g/m²/y. Measured rate of thickness loss may also be reported in mm/y or μm/y, but shall only be reported for alloys that uniformly corrode, and shall not be reported for alloys with localized corrosion such as pitting or intergranular corrosion.

3.11 Time based measurements of corrosion rate can be integrated to obtain estimates of cumulative charge passed or cumulative mass loss. Cumulative charge or mass loss shall be expressed as coulombs (C/m²) or grams (g/m²), respectively. Measures of thickness loss may also be reported (mm or μm), but shall only be reported for alloys that uniformly corrode. When cumulative mass loss is reported, cumulative mass loss should be correlated to a reference material mass loss.¹

3.12 Coating Use – Free Corrosion Sensors: For use in coating tests, the free corrosion sensor should be flush mounted to form a planar surface that is greater than the sensor gage area along each edge (Figures A1 and A2). This may be achieved by casting, potting, or mounting the sensors. The potting materials shall be environmentally resistant, and protect and electrically isolate the electrode surfaces and electrical connections, other than the sensing areas. Planarity is important to maintaining a continuous uniform coating thickness over the sensing area.

3.12.1 The free corrosion sensor responses are dependent on coating properties and coating defect area. The sensor response will change during the test as coating degradation progresses. Therefore, the measured current is a nominal measurement of overall response, and may not be a measure of uniform current distribution over the surface of an electrode.

3.12.2 Current range required for a measurement of coating performance will depend on the coating properties, presence of a defect, defect geometry, sensor alloy, electrode area, amplitude of excitation voltage, and test environment. The measurement corrosion rate range will be dependent on the expected corrosion rates of the given sensor alloy.

3.12.3 The current measured with the coated free corrosion sensor is an indicator of the instantaneous corrosion rate dependent on existing material and environmental conditions and not a measure of absolute corrosion rates.

3.12.4 For coated free corrosion sensors, 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 – 10 Hz. The average of multiple cycles should be used to obtain the current measurement.

3.12.5 Current shall be reported as total current (I_c) and shall be measured in milliamperes (mA).

3.12.5.1 Total current is used for coating evaluations because the active area of the electrode is difficult to define since it may be dominated by coating nonuniformity and defects.

3.12.6 Time based measurements of current can be integrated to obtain estimates of cumulative charge passed. Cumulative charge shall be expressed as coulombs (C).

3.13 Preparation – Free Corrosion Sensors: The free corrosion sensors shall be prepared and cleaned as specified by the sensor supplier. Mechanical surface preparation should be avoided for thin film or foil sensing elements. Composite

laminated sensors with sufficient electrode thickness, approximately greater than 1 mm, may be mechanically finished using abrasives such as 600-grit sandpaper.

3.13.1 For sensors used in the bare condition, without coatings, the sensors shall be cleaned to remove soluble organic and inorganic contaminants. Cleaning chemicals and processes need to be compatible with the sensor electrode and dielectric materials.

3.13.2 The electrochemical sensors may be used to evaluate the protective properties of surface treatments and coatings.¹⁰ The range of protective materials that may be used with the electrochemical sensors is very broad and specific requirements may need to be established for each. Below is a general description of the use of electrochemical sensors with an organic coating system that may consist of pretreatment, primer, and topcoat. Besides traditional paints and coatings, other materials that may be used include chemical conversion coatings, vapor phase inhibitors, protective oils, and corrosion preventative compounds.

3.13.2.1 Sensors to be used with organic coatings may be prepared using chemical cleaning and pretreatment processes required for the specific application of interest. Coating performance is strongly dependent on surface cleanliness and preparation. Some high temperature and chemical process steps such as descaling, deoxidation, and desmutting may damage the dielectric materials and sensor electrodes. The as-received sensors should be in a relatively clean contaminant-free condition. Metal products with, for example mill finish surfaces, might require a more aggressive cleaning process. Given these material conditions, and other unique cleaning and pretreatment requirements, some process modifications may be required for use of the electrochemical sensors. It is recommended that cleaning and pretreatment process be validated on a set of replicate sensors prior to initiating full scale coating tests with the electrochemical sensors. The sensors should be inspected after each step of the coating process.

3.13.2.2 Sensors used to test coatings should be processed, as much as possible, like typical parts. Coatings can be applied by spray, brush, or dip processes. Electrical contacts and connectors shall be masked, or otherwise protected from the coating processes. Thermally cured coatings may be applied to the sensors, but resistance of the sensor to process temperatures shall be verified. Coatings should be applied and cured according to manufacturer's specifications. Specific coating properties such as dry film thickness, adhesion, and cure properties should be tested on witness panels processed at the same time as the sensor electrodes.

3.13.2.3 Coating defects may be produced on free corrosion sensors using masking or scribing techniques.¹⁰ The defects should be oriented transverse to the interdigitated electrodes, and maybe oblique or normal to the electrodes (Appendix A, Figure A3). One or more defects may be applied to each sensor.

3.13.2.3.1 Coating defects may be formed by masking the sensor using thin strips of tape that extend across the full width of the sensing element.

3.13.2.3.2 Mechanical scribing of thick composite laminated electrodes may be done using standard manual or automated methods for coated panels.⁹ Mechanical scribing shall not be performed on deposited or thin film laminated electrodes.

Scribes shall fully penetrate the coating to the substrate, and have a uniform width that should be 0.5 to 1.5 mm. Tearing and delamination of the coating at the scribe edge should be minimized. After mechanical scribing, the impedance shall be assessed to demonstrate that the electrodes are not electrically shorted by metal smearing or debris in the scribe (Paragraph 3.14.3.2). If an electrical short is detected, the scribe should be dressed with a fine grit silicon carbide paper or with an abrasive file. Fine grit sand paper should be 800 to 1,000-grit for the purposes of dressing the scribe.

3.14 Specification and Inspection – Free Corrosion Sensors: Sensors should be inspected prior to sensor processing and before use in atmospheric corrosion tests.

3.14.1 Sensors shall be visually inspected for any irregularities such as defects or pores in the dielectric materials, damage to the electrodes, irregular geometry, electrode defects, surface contaminants, or other sensor-to-sensor anomalies that may affect performance and consistency of results.

3.14.2 Sensor range and span for free corrosion rate shall be determined either theoretically or experimentally before conducting exposure tests. The nominal expected span for the sensors should be established for a representative environment and the expected test conditions.

3.14.3 Verification of sensor condition shall be done by electrical measurements.

3.14.3.1 Continuity between the gage area of the electrodes and sensor connector should be checked. Care should be taken not to scratch or otherwise damage the test surface during this measurement.

3.14.3.2 Impedance measurements between the two electrodes of the IDE sensor shall be made on the as-produced or as-received sensors and shall be repeated again immediately prior to testing after preparation is complete. Impedance measurements may be done using the measurement instrument excitation, full spectrum EIS measurement, or DC resistance meter.^{8,11} This test is done to insure that the two electrodes are electrically isolated from each other prior to use. The DC resistance shall be greater than 100 MΩ. Impedance measurements shall be performed in environmentally controlled conditions with RH less than 50% and temperature between 20 °C and 27 °C.

3.14.3.3 Current measurements using the sensor and measurement system instrument should be verified using known resistances or dummy cells that load the excitation circuitry within the stated sensor span.

3.14.3.4 The temperature dependence of the sensing element shall be determined in controlled temperature and constant humidity (<50% RH) tests. The temperature range tested should cover the specified operational range of the sensor and that range shall be stated. These temperature tests may be performed at other humidity levels (30% < RH < 95%).

3.14.4 Sensor performance shall be verified in reference solutions.

3.14.4.1 Sensors used to verify performance should not be reused for atmospheric testing.

3.14.4.2 Free corrosion sensors may be tested by immersing in known electrolytes

3.14.4.2.1 Free corrosion sensors shall be immersed in salt solution at a known temperature and excited using either a full spectrum electrochemical impedance spectroscopy (EIS) measurement or at the measurement frequency.

3.14.4.2.2 The time between exposure and testing should be noted, and the time shall be sufficient for the corrosion sensor to reach a steady state condition.

3.14.4.2.3 Test conditions, such as solution chemistry, concentration, and temperature may be application specific. It is recommended that three conditions be tested that produce sensor response in each third (low, midrange, and high) of the stated corrosion rate range.

Section 4: Galvanic Corrosion Sensors

4.1 Galvanic corrosion measurements are obtained using two-electrode sensors with the electrodes connected together using either: 1) a zero resistance ammeter, or 2) a precision resistor (Figures A1 and A2).^{6,12-14}

4.2 Two-electrode sensors for measurement of galvanic corrosion can be fabricated from any alloys of interest for a given application or reference alloys for determining the relative severity of a given environment. The electrodes of the two-electrode sensor shall be constructed of dissimilar materials to form a galvanic couple.

4.3 The separation distance between the electrodes should be uniform and no more than 300 μm . A high electrode digit length to width ratio may minimize contribution of edge effects that distort current and potential distributions, and small width electrodes support 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.

4.4 Geometry of the electrodes and cathode to anode area ratio will influence the spatial distribution and magnitude of the galvanic current. The area ratio may be selected based on the known cathode and anode areas for a particular application.

4.5 Galvanic current range will depend on the alloys, sensing area of each alloy electrode, and test environment. Spans for current measurements should be selected based on these factors. The galvanic corrosion measurement range (maximum and minimum measurements) and span (difference in maximum and minimum full-scale reading) will be dependent on the expected galvanic corrosion rates of the given sensor alloys.

4.6 Electrical measurement of the galvanic current may be done using either of two methods: 1) zero resistance ammeter, or 2) precision resistor measurements.

4.6.1 Method 1 – Zero Resistance Ammeter. Galvanic sensor excitation shall be done using a ZRA that applies a current bias to control the potential difference between the electrodes of the galvanic couple to zero. The current required to achieve this potential control is the galvanic current.

4.6.1.1 During the time interval between active ZRA measurements, the two electrodes of the sensor shall be controlled to the same potential or electrically shorted.

4.6.2 Method 2 – Precision Resistor. Galvanic current shall be determined by measuring the voltage drop across a precision resistor that connects the two alloys of the galvanic couple. Knowing the voltage drop (V) and the value of the precision resistor (R), the galvanic current (I) can be calculated using Ohm's Law ($I = V/R$). The resistor produces a finite voltage difference between the anode and cathode, and the resistor value should be as low as possible, while still allowing the current measurement to meet the precision requirements.

4.6.2.1 Between measurements, the two electrodes of the sensor shall be electrically connected through the precision resistors.

4.7 Galvanic corrosion current (i_g) shall be reported as mA/cm². Current density will be measured using the area of the anode (more electronegative alloy).

4.8 Corrosion rate may be converted to mass loss rate using Faraday's Law.⁹ Mass loss rate shall be expressed as g/m²/y. Measured rate of thickness loss may also be reported in mm/y or μ m/y, but shall only be reported for alloys that uniformly corrode, and shall not be reported for alloys with localized corrosion such as pitting or intergranular corrosion.

4.9 Time based measurements of corrosion rate can be integrated to obtain estimates of cumulative mass loss. Cumulative mass loss shall be expressed as g/m². Measures of thickness loss may also be reported (mm or μ m), but shall only be reported for alloys that uniformly corrode. When cumulative mass or thickness loss is reported, cumulative damage should be correlated to a reference material.¹

4.10 Coating Use – Galvanic Corrosion Sensors: For use in coating tests, the sensor should be flush mounted to form a planar surface that is greater than the sensor gage area along each edge (Figures A1 and A2). This may be achieved by casting, potting, or mounting the sensors. The potting materials shall be environmentally resistant, and protect and electrically isolate the electrode surfaces and electrical connections, other than the sensing areas. Planarity is important to maintaining a continuous uniform coating thickness over the sensing area.

4.10.1 The galvanic corrosion sensor responses are dependent on coating properties and coating defect area. The sensor response will change during the test as coating degradation progresses. Therefore, the corrosion current is a nominal measurement of overall response, and may not be a measure of uniform current distribution over the surface of an electrode.

4.10.2 Galvanic current range required for a measurement of coating performance will depend on the coating properties, presence of a defect, defect geometry, sensor materials, electrode areas, and test environment. The measurement range will be dependent on the expected galvanic corrosion rates of the given sensor alloy.

4.10.3 Electrical measurement of the galvanic current may be done using either zero resistance ammeter or precision resistor measurements (Paragraph 4.6).

4.10.4 Galvanic corrosion shall be reported as total galvanic current (I_g) and shall be measured as mA.

4.10.4.1 Total current is used for coating evaluations. The active area of the electrode is difficult to define, since it may be dominated by coating nonuniformity and defects.

4.10.5 Galvanic corrosion measurements may be converted to mass loss rate using Faraday's Law.⁹ Mass loss rate shall be expressed as g/y.

4.10.6 Time based measurements of galvanic corrosion rate can be integrated to obtain estimates of cumulative charge passed or cumulative mass loss. Cumulative charge or mass loss shall be expressed as coulombs (C) or grams (g), respectively. When cumulative mass loss is reported, cumulative damage should be correlated to a reference material.¹

4.11 Preparation – Galvanic Corrosion Sensors: The galvanic sensors shall be prepared as specified by the sensor supplier. Mechanical surface preparation should be avoided for thin film or foil sensing elements. Composite laminate sensors with sufficient electrode thickness, approximately greater than 1 mm, may be mechanically finished using abrasives such as 600-grit sandpaper.

4.11.1 For sensors used in the bare condition, without coatings, the sensors shall be cleaned to remove soluble organic and inorganic contaminants. Cleaning chemicals and processes need to be compatible with the sensor electrode and dielectric materials.

4.11.2 The electrochemical sensors may be used to evaluate the protective properties of surface treatments and coatings.¹⁰ The range of protective materials that may be used with the electrochemical sensors is very broad and specific requirements may need to be established for each. Below is a general description of the use of electrochemical sensors with an organic coating system that may consist of pretreatment, primer, and topcoat. Besides traditional paints and coatings, other materials that may be used include chemical conversion coatings, vapor phase inhibitors, protective oils, and corrosion preventative compounds.

4.11.2.1 Sensors to be used with organic coatings may be prepared using chemical cleaning and pretreatment processes required for the specific application of interest. Coating performance is strongly dependent on surface cleanliness and preparation. Some high temperature and chemical process steps such as descaling, deoxidation, and desmutting may damage the dielectric materials and sensor electrodes. The as-received sensors should be in a relatively clean contaminant free condition. Metal products with, for example mill finish surfaces, might require a more aggressive cleaning process. Given these material conditions, and other unique cleaning and pretreatment requirements, some process modifications may be required for use of the electrochemical sensors. It is recommended that cleaning and pretreatment process be validated on a set of replicate sensors prior to initiating full scale coating tests with the electrochemical sensors. The sensors should be inspected after each step of the coating process.

4.11.2.2 Sensors used to test coatings should be processed, as much as possible, like typical parts. Coatings can be applied by spray, brush, or dip processes. Electrical contacts and connectors shall be masked, or otherwise protected from the coating processes. Thermally cured coatings may be applied to the sensors, but resistance of the sensor to process temperatures shall be verified. Coatings should be applied and cured according to manufacturer's specifications. Specific coating properties such as dry film thickness, adhesion, and cure properties should be tested on witness panels processed at the same time as the sensor electrodes.

4.11.2.3 Coating defects may be produced on galvanic corrosion sensors using masking or scribing techniques.¹⁰ The defects should be oriented transverse to the interdigitated electrodes, and maybe oblique or normal to the electrodes (Appendix A, Figure A3). One or more defects may be applied to each sensor.

4.11.2.3.1 Coating defects may be formed by masking the sensor using thin strips of tape that extend across the full width of the sensing element.

4.11.2.3.2 Mechanical scribing of thick composite laminate electrodes may be done using standard manual or automated methods for coated panels.¹⁰ Mechanical scribing shall not be performed on deposited or thin film laminate electrodes. Scribes shall fully penetrate the coating to the substrate, and have a uniform width that should be 0.5 to 1.5 mm. Tearing and delamination of the coating at the scribe edge should be minimized. After mechanical scribing, the impedance shall be assessed to demonstrate that the electrodes are not electrically shorted by metal smearing or debris in the scribe (Paragraph 4.12.3.2). If an electrical short is detected, the scribe should be dressed with a fine grit silicon carbide paper or with an abrasive file. Fine grit sand paper should be 800 to 1,000-grit for the purposes of dressing the scribe.

4.12 Specification and Inspection – Galvanic Corrosion Sensors: Sensors should be inspected prior to sensor processing and before use in atmospheric corrosion tests.

4.12.1 Sensors shall be visually inspected for any irregularities such as defects or pores in the dielectric materials, damage to the electrodes, irregular geometry, electrode defects, surface contaminants, or other sensor-to-sensor anomalies that may affect performance and consistency of results.

4.12.2 Sensor range and span for galvanic corrosion rate shall be determined either theoretically or experimentally before conducting exposure tests. The nominal expected span for the sensors should be established for a representative environment and the expected test conditions.

4.12.3 Verification of sensor condition shall be done by electrical measurements.

4.12.3.1 Continuity between the gage area of the electrodes and sensor connector should be checked. Care should be taken not to scratch or otherwise damage the test surface during this measurement.

4.12.3.2 Impedance measurements between the two electrodes of the IDE sensor shall be made on the as produced or as received sensors and shall be repeated again immediately prior to testing after preparation is complete. Impedance measurements may be done using the measurement instrument excitation, full spectrum EIS measurement, or DC resistance meter.^{8,11} This test is done to insure that the two electrodes are electrically isolated from each other prior to use. The DC resistance shall be greater than 100 MΩ. Impedance measurements shall be performed in environmentally controlled conditions with RH less than 50% and temperature between 20 °C and 27 °C.

4.12.3.3 Galvanic current measurements using the sensor and measurement system instrument should be verified using known current sources that load the excitation and acquisition circuitry within the stated sensor span.

4.12.3.4 For the galvanic current Method 1 – Zero Resistance Ammeter (Paragraph 4.6.1), the instrument specification should include a statement of precision for electrochemical potential difference control between the two electrodes.

4.12.3.5 For the galvanic current Method 2 – Precision Resistor (Paragraph 4.6.2), the instrument specification should include the resistance value between the two electrodes and precision of the resistance.

4.12.3.6 The temperature dependence of the sensing element shall be determined in controlled temperature and constant humidity (<50% RH) tests. The temperature range tested should cover the specified operational range of the sensor and that range shall be stated. These temperature tests may be performed at other humidity levels (30% < RH < 95%).

4.12.4 Sensor performance shall be verified in reference solutions prior to use.

4.12.4.1 Sensors used to verify performance shall not be reused for atmospheric testing.

4.12.4.2 Galvanic sensors shall be tested by immersing in known electrolytes.

4.12.4.2.1 Galvanic corrosion sensors shall be immersed in a known salt solution at a controlled temperature.

4.12.4.2.2 The galvanic current should be recorded for a period sufficient to reach a relatively steady state current response.

4.12.4.2.3 Test conditions, such as solution chemistry, concentration, and temperature may be application specific. It is recommended that three conditions be tested that produce sensor response in each third (low, midrange, and high) of the stated galvanic corrosion rate range.

Section 5: Surface Conductance Sensors

5.1 Conductance measurements can be used to measure moisture and contaminants that form electrolyte layers on the sensor surface. The hygroscopic properties of the contaminant deposits such as salts will affect the moisture layer and conductance measurements. The conductance measurement is indicative of conditions that may promote corrosion. Conductance measurements are obtained using two-electrode sensors.

5.2 Interdigitated two-electrode sensors produced from noble metals and alloys, such as gold, shall be used for surface conductance measurements.

5.3 The separation distance between the electrodes should be uniform and no more than 300 µm. A high electrode digit length to width ratio may minimize contribution of edge effects that distort current and potential distributions, and small width electrodes support 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.

5.4 Electrical excitation of sensors shall be done by applying a voltage signal between the two electrodes. During measurements, the relative potential between the two electrodes is controlled and current response recorded.

5.5 Current range will depend on the electrode test area, excitation voltage, and test environment. Spans for current measurements used to determine conductance should be selected based on these factors. The conductance measurement range (maximum and minimum measurements) and span (difference in maximum and

minimum full-scale reading) will be dependent on the expected contaminant composition and amount deposited on the sensor surface.

5.6 Between measurements, the two electrodes of the sensor should be electrically shorted.

5.7 Current response shall be measured using voltage sine wave excitation. Excitation frequency shall be within the range of 0.1 kHz – 1,000 kHz for conductance sensors used in the bare condition.

5.8 The excitation signal shall be a voltage sine wave with amplitude between the two electrodes of not more than 30 mV. The conductance shall be measured as the ratio of the I_{rms}/V_{rms} .

5.9 Conductance shall be measured as Siemens (S).

5.10 Coating Use – Conductance Sensors: For use in coating tests, the sensors should be flush mounted to form a planar surface that is greater than the sensor gage area along each edge (Figures A2 and A3). This may be achieved by casting, potting, or mounting the sensors. The potting materials shall be environmentally resistant, and protect and electrically isolate the electrode surfaces and electrical connections other than the sensing areas. Planarity is important to maintaining a continuous uniform coating thickness over the sensing area.

5.10.1 Conductance measurements with interdigitated, noble metal, two-electrode sensors are used to measure protective properties of coatings applied to the sensor. The two-electrode conductance sensors shall be used in the fully coated condition for measuring coating properties.

5.10.2 Conductance measurement range required for a measurement of coating performance will depend on the coating properties, electrode area, amplitude of excitation voltage, and test environment.

5.10.3 Current response shall be measured using voltage sine wave excitation. Single or multiple frequency measurements may be used to characterize coating condition. Excitation frequency should include the range from 0.1 Hz – 1,000 Hz. The frequency measurement duration should be sufficient to obtain a stable response.

5.10.4 The excitation signal shall be a sine wave with a voltage amplitude between the two electrodes of not more than 30 mV. The conductance shall be measured as the ratio of the I_{rms}/V_{rms} .

5.10.5 Conductance shall be measured as Siemens (S).

5.11 Preparation – Conductance Sensors: Noble metals and alloys, such as gold, shall be used for the electrode material of the conductance sensor.

5.11.1 For sensors use in the bare condition, without coating, the sensors shall be cleaned to remove organic and inorganic contaminants. An example cleaning procedure is wiping using oil-free swabs or wipes with isopropyl alcohol, followed by deionized water rinsing, and hot air drying. Cleaning chemicals and processes need to be compatible with the sensor electrode and dielectric materials.

5.11.2 The electrochemical sensors may be used to evaluate the protective properties surface treatments and coatings.¹⁰ The range of protective materials that may be used with the electrochemical sensors is very broad and specific requirements may need to be established for each. Below is a general description

of the use of electrochemical sensors with an organic coating system that may consist of pretreatment, primer, and topcoat. Besides traditional paints and coatings, other materials that might be used may include chemical conversion coatings, vapor phase inhibitors, protective oils, and corrosion preventative compounds.

5.11.2.1 Sensors to be used with organic coatings may be prepared using chemical cleaning and pretreatment processes required for the specific application of interest. Coating performance is strongly dependent on surface cleanliness and preparation. Some high temperature and chemical process steps such as descaling, deoxidation, and desmutting may damage the dielectric materials and sensor electrodes. The as-received sensors should be in a relatively clean contaminant free condition. Metal products with, for example mill finish surfaces, might require a more aggressive cleaning process. Given these material conditions, and other unique cleaning and pretreatment requirements, some process modifications may be required for use of the electrochemical sensors. It is recommended that cleaning and pretreatment process be validated on a set of replicate sensors prior to initiating full scale coating tests with the electrochemical sensors. The sensors should be inspected after each step of the coating process.

5.11.2.2 Sensors used to test coatings should be processed like typical parts. Coatings can be applied by spray, brush, or dip processes. Electrical contacts shall be masked, or otherwise protected from becoming coated. Thermally cured coatings may be applied to the sensors, but temperature stability of the materials of construction shall be specified for the intended use and resistance of the sensor to process temperatures shall be verified. Coatings should be applied and cured according to manufacturer's specifications. Specific coating properties of such as dry film thickness, adhesion, and cure properties should be tested on witness panels processed at the same time as the sensor electrodes.

5.11.2.3 The conductance sensors shall only be tested with intact coatings with no applied artificial defects.

5.12 Specification and Inspection – Conductance Sensors: Sensors shall be inspected prior to sensor processing and use for testing.

5.12.1 Sensors shall be visually inspected for any irregularities such as defects or pores in the dielectric materials, damage to the electrodes, irregular geometry, electrode defects, surface contaminants, or other sensor-to-sensor anomalies that may affect performance and consistency of results.

5.12.2 Sensor range and span for conductance shall be determined either theoretically or experimentally before conducting exposure tests. The nominal expected span for the sensors should be established for a representative environment and the expected test conditions.

5.12.3 Verification of sensor condition shall be done by electrical measurements.

5.12.3.1 Continuity between the gage area of the electrodes and sensor connector should be checked. Care should be taken not to scratch or otherwise damage the test surface during this measurement.

5.12.3.2 Impedance measurements between the two electrodes of the IDE sensor shall be made on the as-produced or as-received sensors and shall be repeated again immediately prior to testing after preparation is complete. Impedance measurements may be done using the measurement instrument excitation, full spectrum EIS measurement, or DC resistance meter. This test

is done to insure that the two electrodes are electrically isolated from each other prior to use. The DC resistance shall be greater than 100 MΩ. Impedance measurements shall be performed in environmentally controlled conditions with RH less than 50% and temperature between 20 °C and 27 °C.

5.12.3.3 Conductance measurements using the sensor and measurement system instrument should be verified using known resistances or dummy circuits that load the excitation circuitry within the stated sensor span.

5.12.3.4 The temperature dependence of the sensing element shall be determined in controlled temperature and constant humidity (<50% RH) tests. The temperature range tested should cover the specified operational range of the sensor and that range shall be stated. These temperature tests may be performed at other humidity levels (30% < RH < 95%).

5.12.4 Sensor performance shall be verified in reference solutions prior to use.

5.12.4.1 Sensors used to verify performance shall not be reused for atmospheric testing.

5.12.4.2 Conductance sensors shall be tested by immersing in standard conductivity electrolytes.

5.12.4.2.1 Conductance sensors shall be immersed in standard conductivity electrolytes at a known temperature and excited using either a full spectrum EIS measurement or at the measurement frequency.

5.12.4.2.2 The time between exposure and testing should be noted, and the time should be sufficient for the sensor to reach equilibrium with the test environment.

5.12.4.2.3 Test conditions, such as solution chemistry, concentration, and temperature may be application specific. It is recommended that three conditions be tested that produce sensor response in each third (low, midrange, and high) of the stated sensor conductance measurement range.

Section 6: Atmospheric Testing

6.1 The electrochemical sensors can be used in accelerated laboratory tests, outdoor exposures, and service environments.

6.2 The sensors should be arranged and oriented using the same practices used for other standard test panels.¹⁰

6.3 Longevity of an electrochemical sensor will depend on aggressiveness of the environment, presence of a coating, coating defect, and durability of the sensing elements.

6.4 Sensor selection should consider expected accelerated test or exposure environment severity.^{15,16}

6.4.1 It may be necessary to run preliminary tests to verify that the sensors provide the longevity and measurement span required for the given environment.

6.5 The sampling interval is dependent on the rate of change of the exposure conditions.

Based on test requirements, faster or slower sampling intervals may be used. Cyclic tests that impose rapidly changing conditions may require sampling intervals of a few minutes; while sampling intervals of tens of minutes to an hour may be acceptable for long-term outdoor exposure tests.

- 6.6** All data shall be time and date stamped using Coordinated Universal Time (UTC) conventions. For outdoor testing and evaluation of mobile assets, UTC +0:00 shall be used. Clocks shall be verified versus UTC prior to initiating a test. Clock accuracy for the specific electronics should be stated as part of the system specifications and reporting.

Section 7: Reporting

- 7.1** Guidance is given for reporting sensor information, results, and environmental test parameters (Appendix C [nonmandatory]),^{17,18} The types of sensor used shall be identified: free corrosion, galvanic corrosion, or conductance. Reporting shall include sensor specific information including sensor serial number, electrode materials of construction, geometry, and electrode areas. Sensor output spans and data sampling rates shall be stated. For free corrosion and galvanic corrosion sensors, the method used to determine corrosion rate shall be identified (Paragraphs 3.7 and 4.6).
- 7.2** Surface preparation products and material identification shall be reported along with any observational or quantitative data related to the sensor inspection, condition, cleaning, pretreatment, and coating process conditions and results. When applicable, coating information including types of coatings, application methods, and curing/drying schedule shall be reported. When witness panels are processed with the electrochemical sensors for coating characterization, such as degree of cure, adhesion, film thickness etc., those data should be included. If the coated samples were prepared with artificial defects, the method and tools used to produce the defect shall be reported.
- 7.3** Reporting shall include identification of the type of test performed, start date, end date, and duration. Reporting shall include notes on any upset conditions or other details that may affect the results of the atmospheric corrosion tests. If environmental and quality control data (mass loss, collection rates, chemistry, etc.) are collected during the exposure tests, these data should be included in the report or reference shall be made to these records to allow traceability.
- 7.4** The electrochemical sensor data shall be stored in a digital format such as comma separated variable (.csv) text files. The first column of each file will contain a time stamp (UTC). Depending on the type of sensors used and quantity of sensors, additional data columns should be added. Each column will include header information for unique identification of the sensor, data type, and units. Data may include corrosion rate, cumulative corrosion, and conductance. The digital data sheet may also include environmental measures of sensor surface temperature, air temperature, and relative humidity.¹⁹ Relevant environmental data from other sources such as time based records of test chamber conditions or environmental data from monitoring stations should be include with the digital test records.

Section 8: Precision and Bias

- 8.1** The performance of the electrochemical measurement system will be dependent on the sensors, preparation process, and supporting electronics. Electrical measurements shall be used to obtain precision and bias statements for the given sensor and measurement systems as described for each type of sensor in Para-

graphs 3.12, 4.12, and 5.12. The sensors and measurements described in this test method produce outputs that are related to corrosion rate and surface electrolyte conductivity. Precision and bias of each type of electrochemical sensor and measurement method for estimating corrosion rate and cumulative corrosion should be validated for the given materials of construction and environment of interest relative to ground truth mass loss, volume loss, damage depth, or coating condition measurements. Prescriptive description of these processes for particular environments, sensor alloys, electrode geometries, and applications are beyond the scope of this standard, but guidance may be obtained through guides for performing mass loss measurements and conducting statistical data analysis.^{1,20}

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Appendix A

Example Images of Electrochemical Sensors (Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

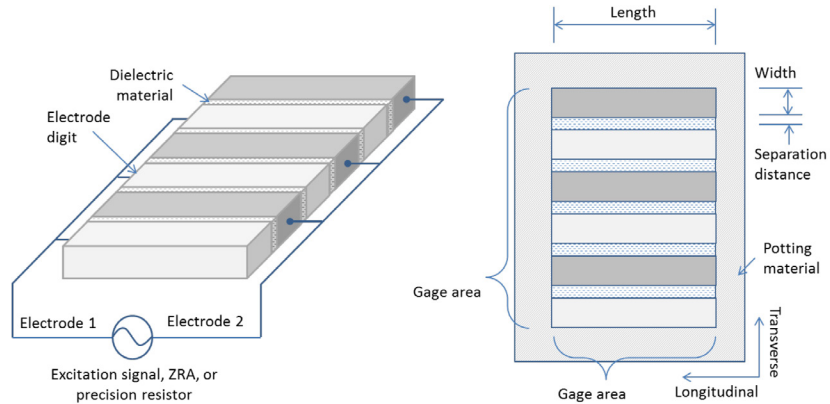


FIGURE A1: Schematic Isometric and Top View of Laminated Interdigitated Two-Electrode Sensor. The two electrodes are formed by shorting together every other digit to form the interdigitated electrodes. Each electrode is made of a single alloy or metal, but the two electrodes may be either the same material (free corrosion sensor) or different materials (galvanic corrosion sensor).

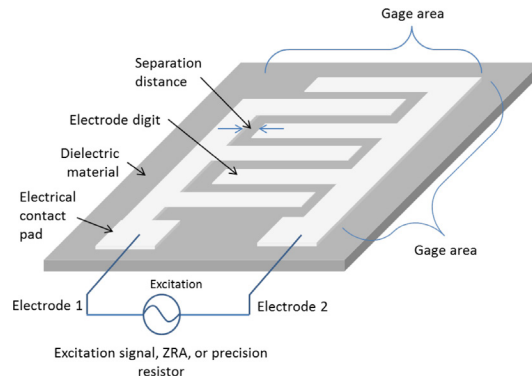


FIGURE A2: Schematic of Laminated Thin Film Interdigitated Two-Electrode Sensor. The two electrodes are formed by patterning the thin film deposit or bonded foil on the dielectric material. Each electrode is made of a single alloy or metal, but the two electrodes may be either the same material (free corrosion sensor) or different materials (galvanic corrosion sensor).

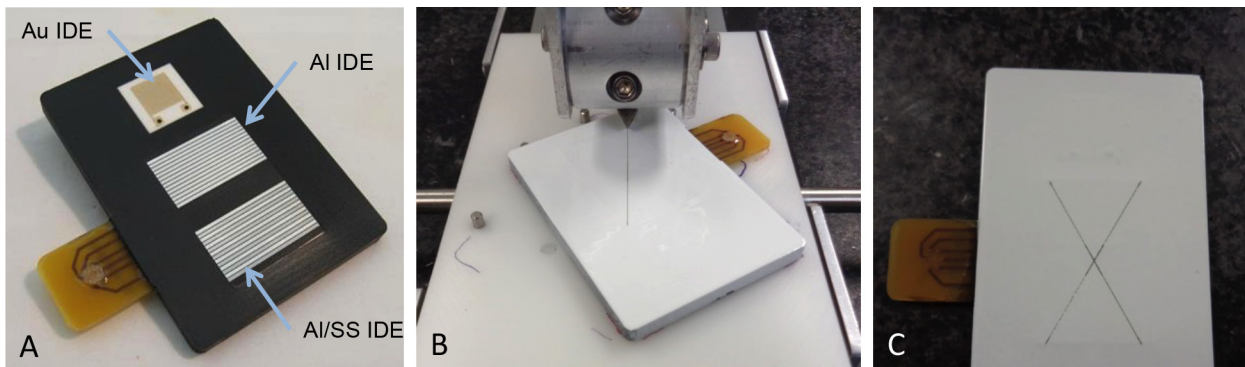


FIGURE A3: Figure A3: Electrochemical Sensors for Free Corrosion, Galvanic Corrosion, and Conductance in a Single Panel (A). The panel dimensions are approximately 5 cm by 9 cm. Example of Painted Sensors with Free Corrosion and Galvanic Corrosion Sensor Scribed (B and C).²¹

Appendix B Example of Reporting Information (Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

The measurement of polarization resistance is for bare sensors only and not for use with coatings and coated sensors. Polarization resistance (R_p) can be used to calculate the corrosion rate (I_{corr}) of a metal or alloy using the Stern-Geary Equation.⁷

$$R_p = \frac{(\Delta E)}{(\Delta I)_{\Delta E \rightarrow 0}} = \frac{\beta_a \beta_c}{2.3 I_{corr} (\beta_a + \beta_c)}, \text{ or } \frac{\beta}{I_{corr}}, \text{ so that, } I_{corr} \propto \frac{1}{R_p} \quad (B1)$$

where β_a and β_c are empirical anodic and cathodic Tafel values, respectively. Experimentally, R_p can be obtained by measuring the resistance of a two-electrode circuit with each working electrode fabricated from the same material with equal areas (WE_1 and WE_2). The simple equivalent circuit for this two-electrode arrangement consists of resistive and capacitive elements (Figure B1). The impedance of the equivalent circuit at low frequency excitation ($\omega \rightarrow 0$), and high frequency ($\omega \rightarrow \infty$) are:

$$Z_{\omega \rightarrow 0} = 2R_p + R_s, \text{ and } Z_{\omega \rightarrow \infty} = R_s, \text{ respectively} \quad (B2)$$

where R_s is the solution resistance; therefore:

$$I_{corr} = \frac{2\beta}{Z_{\omega \rightarrow 0} - R_s} \quad (B3)$$

or

$$R_p = \frac{Z_{\omega \rightarrow 0} - R_s}{2} \quad (B4)$$

In general, R_s is much less than the polarization resistance this further simplifies the measurement and calculation of I_{corr} . More precise measurements of R_p may be possible by measuring R_s using a higher frequency excitation signal (1 to 10 kHz).

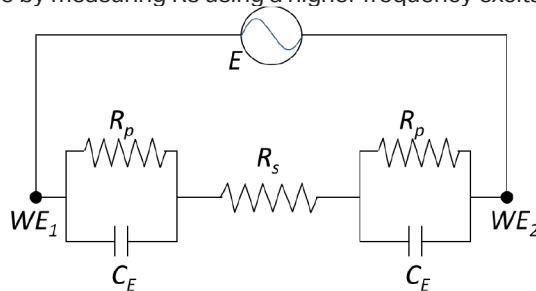


FIGURE B1: Equivalent Circuit for Two-Electrode Corrosion Measurement

Appendix C

Example of Reporting Information (Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

Results reporting information

1. Test
 - a. Report date
 - b. Report author
 - c. Test operator
 - d. Test No.
 - e. Type/Location
 - i. Local time zone UTC ± XX:XX
 - f. Environment description
 - g. Chemistry/composition/concentration
 - h. Schedule
 - i. Start Date
 - ii. Scheduled End Date
 - iii. Actual End Date
 - iv. Outages
 - i. Duration
2. Sensor
 - a. Manufacturer
 - b. Model No.
 - c. Serial No.
 - d. Calibration date
 - e. Type
 - f. Geometry
 - g. Material(s)
 - h. Test condition (bare, coated, etc.)
 - i. Preparation processes
 - j. Sensor span
 - k. Data collection rates
3. Coatings
 - a. Cleaner(s)
 - b. Pretreatment
 - c. Coating(s)

Electrochemical sensor system output format

1. Time (UTC)
2. Corrosion rate (free or galvanic)
3. Cumulative corrosion (free or galvanic)
4. Conductance

Environmental Parameters

1. Time (UTC)
2. Surface Temperature
3. Air Temperature
4. Relative Humidity (%RH)
5. Contaminant deposition rates
6. Rainfall

