Designation: D8370 - 22

Standard Test Method for Field Measurement of Electrochemical Impedance on Coatings and Linings¹

This standard is issued under the fixed designation D8370; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

- 1.1 This test method covers the procedure for field measurement of electrochemical impedance spectroscopy (EIS) for polymeric coatings over conductive substrates.
- 1.2 This test method covers the parameters for determining an adequate sample size.
- 1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents at a log/standards

- 2.1 ASTM Standards:²
- D16 Terminology for Paint, Related Coatings, Materials, and Applications
- D610 Practice for Evaluating Degree of Rusting on Painted Steel Surfaces
- D660 Test Method for Evaluating Degree of Checking of Exterior Paints
- D661 Test Method for Evaluating Degree of Cracking of Exterior Paints
- D714 Test Method for Evaluating Degree of Blistering of Paints
- ¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.48 on Durability of Pipeline Coating and Linings.
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- ² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G106 Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements
- G193 Terminology and Acronyms Relating to Corrosion
- G215 Guide for Electrode Potential Measurement
- 2.2 ISO Standards:³
- ISO 16773-1 Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens—Part 1: Terms and definitions
- ISO 16773-2 Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens—Part 2: Collection of data
- ISO 16773-3 Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens—Part 3: Processing and analysis of data from dummy cells
- ISO 16773-4 Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens—Part 4: Examples of spectra of polymer-coated and uncoated specimens

3. Terminology

- 3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminologies G193 and D16.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *test area, n*—total area of the coating surface within a pair of test cells.
- 3.2.2 *test location*, *n*—discrete physical location(s) selected on each structure for performing the electrochemical impedance spectroscopy (EIS) measurements.

4. Significance and Use

- 4.1 This test method is suitable for in-service condition assessment and quality control (QC) testing.
- 4.2 This technique is used to investigate a polymer barrier coating over a conductive substrate and is limited to exposed and accessible coating surfaces.

³ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

- 4.3 This test method is applicable to polymer barrier coatings of all thicknesses provided the impedance is within equipment capabilities. Special considerations are required for evaluation of coatings exceeding 2 mm thickness or containing conductive media, such as metal pigments and conducting polymers.
- 4.4 This test method provides the experimental method needed to ensure proper application of field EIS testing and reporting of its results. This test method uses two test cells per measurement with no electrical connection to the substrate (1-4) (a deviation from the traditional three-electrode measurement) to prevent the need for electrical connection to the underlying structure.

Note 1—The two-test-cell method measures the impedances beneath the two cells plus the impedance of the path between them. This arrangement has additional risks of false negatives/positives that are not encountered using the traditional three-electrode measurement in which an electrical connection to the substrate is made. For this test method, a false positive is defined as a higher impedance value than is typical for the coating, and a false negative is defined as a lower impedance value than is typical for the coating. A traditional three-electrode measurement in the field is possible, but a reliable electrical connection to the substrate can be challenging and may require damage to an otherwise good coating.

4.5 This test method may be used at any time during the life of a coating system. If used for QC, allow for any manufacturer's recommended cure or drying time unless otherwise agreed upon between the participating parties.

Note 2—The results obtained by using this test method could be used for informed coating maintenance decisions, for example, whether to replace a coating system, and may also be applicable as QC measurement for coating contracts.

- 4.6 The results obtained by using this test method shall not be considered as a means for estimating the structural properties of the underlying structure.
- 4.7 The results obtained by using this test method do not measure the corrosion susceptibility of the underlying structure because it uses two test cells with no electrical connection to the substrate. The open circuit potential and voltage perturbation are applied to the test cell electrode, per 5.3, and not the underlying structure.
- 4.8 The electrochemical impedance measurements shall be interpreted by engineers or technical specialists experienced in the fields of protective coatings and corrosion testing. It is often necessary to use other data such as visual inspection, dry film thickness, and adhesion testing, in addition to electrochemical impedance, to formulate conclusions concerning corrosion activity of the underlying structure or the remaining service life of the coating system. See Test Methods D660, D661, D714, or Practice D610 for more information on coating visual inspection.

5. Apparatus

5.1 *Test Cell*—The measurement shall be performed using a surface area of 25 cm² or otherwise agreed upon within each test cell. The test cell should be a nonconductive material. Use adhesives or other means to ensure the surface area is unchanged during testing.

Note 3—To achieve measurement of high-impedance coatings that are

approaching the limit of the instrument's input impedance, larger surface areas may be needed. Typical test cell materials include a 100 mL plastic beaker with the bottom removed or a short length of plastic pipe such as acrylic, polycarbonate, or PVC. A fast-setting marine or silicone adhesive to secure the test cell to the coating surface is preferred when needed. Application of adhesives, especially those using solvents, may be destructive to the field coating being tested; proper repair procedures need to be developed in advance of testing. Commercial gasket-sealed magnetic test cells (in the case of ferritic steel substrates) and similar approaches that combine the test cell and the electrodes into a single unit are also suitable, provided the conditions of 5.3 are met.

5.2 *Electrolyte Solution*—An electrolyte solution shall be added to the test cell to reduce the resistance between the electrodes and the coating surface. The electrolyte solution resistance shall be sufficiently less than the coating being measured.

Note 4—Tap or bottled water generally meets this requirement but may be treated with table salt to decrease the electrolyte resistance further. Other electrolytes such as conductive gels may be suitable.

5.3 Measurement Electrodes—Measurement electrodes are required to perform the test method. At a minimum, two electrodes are required: one electrode to apply the potentiostat voltage and current to one test cell (working electrode (WE)) and one electrode to sense the voltage and current response in the other test cell (counter electrode (CE)). See Guide G106 for more information.

Note 5—Consult with the potentiostat manufacturer for guidance on their specific electrode configurations and how to perform a two-electrode impedance measurement using their equipment.

5.3.1 Working and Counter Electrodes—The WE and CE shall be the same material, inert, and conductive with a surface area that is sufficiently high to not limit uniform current density.

Note 6—Ensure that the WE and CE surfaces are clean and uncorroded before each test for consistent results. Acceptable materials should have low reactivity such as platinum, stainless steel, or graphite.

- 5.3.2 Reference Electrode (RE) (if Used)—The RE can be used to sense the circuit voltage separately rather than coupling both parameters under a CE. Suitable REs include a silver/silver chloride/potassium chloride reference electrode or saturated calomel electrode (SCE). See Guide G215 for more information.
- 5.4 *Electrical Lead Wires*—Use potentiostat manufacturer supplied cable, which should be electrically shielded, for the potentiostat used.

Note 7—External sources of electrical interference should be avoided or diminished. Sources of interferences can include fluorescent lights, motors, and cellular phones in transmitting mode in close proximity. The leads should all be as far away from one another as possible (the counter lead far from the working lead far from the reference lead). Care should be taken in arranging leads by not coiling or crossing over one another so that stray current or inductance is not introduced in the experiment.

5.5 *Potentiostat*—A potentiostat should have a floating design, that is, the WE is not grounded within the instrument. The minimum parameters are a frequency range of 0.01 Hz to 10 000 Hz, an input impedance of $10^{11} \Omega$, a compliance voltage of 5 V, and a current range of ± 100 pA to ± 10 mA with a current resolution no greater than 0.015 % of the current range (see Appendix X1). The potentiostat may use any power

source, although AC power may produce a source of electromagnetic interference.

Note 8—Maintain the potentiostat in accordance with the manufacturer's instructions. Perform dummy cell testing in accordance with Practice G106 to verify that the measurement outputs are within the manufacturer's reported tolerances. For additional information, see ISO 16773-3.

6. Test Location and Test Area

- 6.1 The test area for each measurement is a polymer coating on a conductive substrate, such as coated infrastructure. The conductive substrate beneath the coating shall have electrical continuity between each pair of measured test cells.
- 6.2 Select an area containing undamaged coating at each test location. Remove mud, dirt, and other contaminants from the coating surface using clean water and scrubbing, as needed, and wipe dry with a clean rag. Inspect the surface for cracks, rust-through, and other defects to ensure the selected test area contains no visible coating flaws.

Note 9—This test method is most easily applied to horizontal or near-horizontal surfaces. The coating surface area in contact with the electrolyte will impact results; maintain electrolyte contact with the coating surface area during the measurement. Poor electrolyte contact with the coating surface will result in false positives. Additionally, a thin film of condensed moisture on the coating surface between cells can create a low-impedance pathway that could result in a false negative; for this reason, it is important to ensure the coating surface is dry.

- 6.3 Each test location requires two test cells to perform the impedance measurement using no electrical connection to the substrate. Secure two test cells to the coating surface and achieve a watertight seal. The two cells shall be separated by a distance not less than 5 cm and not more than the manufacturer-supplied cables will allow.
- 6.4 Add electrolyte solution to the test cells, allowing for recommended cure time if an adhesive is used. Allow the electrolyte solution to hydrate into coating to achieve equilibrium. Hydration time may range from 1 h to more than 24 h and is dependent on the coating type, age, quality, and recent exposures to water immersion or humidity (5). The shortest hydration times apply to aged coatings and those in immersion service. If testing is being specified for QC of a newly applied coating, that is, no previous hydration, the time should be at least 48 h or otherwise agreed upon. If testing is being specified for initial service life readings of a new coating, the hydration time required may be much longer.

Note 10—If sufficient hydration time is not given, initial results will lead to false positives. If the impedance value approaches the instrument upper limit, the coating impedance may be higher than the recorded value, and the data should not be used for the coating itself. Attempts to bring the data back into the measurable range by increasing test cell surface area will require post-processing of the impedance data by an engineer or technical specialist. During testing in high humidity, condensate on the coating surface could result in false negatives. See 7.2.5 for related information on reviewing the open circuit potential.

7. Procedure

7.1 *Test Location Sampling Plan*—Develop a sampling plan before testing to determine how many test locations are needed to perform the evaluation. This could use a regular or random approach but shall include at least six measurements.

- Note 11—Six measurements may not be sufficient to evaluate large surface areas. As a guideline, measure 10 to 20 test locations for structures with less than 100 m^2 of coating surface area. For structures with greater than 1000 m^2 of coating surface area, increase the number of test locations to 30 to 50, as practical.
- 7.2 Impedance Measurement—Place the measurement electrodes within the test cells for the respective test area. Submerge the electrode surface area in the electrolyte solution for each measurement and position so they are not in contact with the coating surface during measurement. A two- or three-electrode measurement may be used with minimal to no difference in the resulting data.

Note 12—The RE, if used, should be allowed to stabilize before use. See 7.2.5.

- 7.2.1 *Two-Electrode Measurement*—One test cell contains the WE and the other test cell contains the CE/RE combined as a single electrode (Fig. 1).
- 7.2.2 *Three-Electrode Measurement*—An alternative is for the CE/RE to be a separate CE and RE (Fig. 2).
- 7.2.3 Connect the electrical lead wires to the measurement electrodes and the potentiostat to complete an electrical circuit that includes the potentiostat, electrical lead wires, electrodes, electrolyte, coating beneath each test cell, and conductive substrate.
- 7.2.4 Program the potentiostat software to perform impedance measurements beginning at 10 000 Hz and ending at 0.1 Hz, or lower, applying at least four measured points per decade. The applied voltage shall be a sine wave at the open circuit potential with root mean squared (rms) amplitude of 0.01 V or higher.

Note 13—Use an end frequency of 0.1 Hz to balance test time and usefulness of the data, but use lower frequencies, for example, 0.01 Hz, for diagnostics and spot checks. The applied voltage amplitude required is dependent on the coating material properties and thickness. In addition, all potentiostats have a minimum current that they can measure. If the measurement does not receive the signal, increase the voltage amplitude. High-impedance coatings may require the rms amplitude to be 0.20 V or higher. An open lead measurement, that is, all leads are disconnected and open to air, may be required to evaluate the minimum current that the potentiostat can measure. See ISO 16773-2, Annex A, or the potentiostat manufacturer's instructions for guidelines on conducting an open lead measurement.

7.2.5 If possible, review the open circuit potential to confirm system circuit continuity before beginning the impedance measurement. An acceptable allowance for potential drift is ± 0.005 V per 20 s. If a stable open circuit potential cannot be achieved, check the measurement electrodes, electrical lead wires, and potentiostat for circuit continuity. If the circuit continuity is intact, additional hydration time may be required to accomplish the impedance measurement.

Note 14—An unstable open circuit potential may be caused by insufficient hydration or charging of a higher barrier coating and may result in false positives.

7.2.6 Perform the impedance measurement.

Note 15—If it is desired, the validity of the measurement can be determined by one of several methods. The preferred method is to review the voltage and current data, which should include the origins, that is, the open circuit potential and 0 A, during each impedance measurement and be symmetric in appearance. An alternative approach is to review the open circuit potential after the impedance measurement to ensure it has not



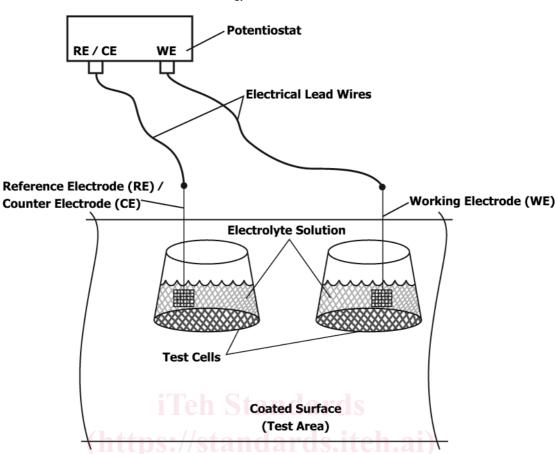


FIG. 1 Two-Electrode Measurement with No Electrical Connection to Substrate

shifted by more than 0.1 V.

Note 16—The temperature at which impedance measurements are recorded should be consistent between measurements. High temperatures, above the glass transition temperature $(T\mathbf{g})$ of the coating, will result in false negatives.

Note 17—The time and number of cycles required for each measurement frequency is generally determined by the instrument software to increase the signal-to-noise ratio to within a desired error.

8. Data Presentation

- 8.1 Raw datasets can be plotted for direct comparison if the surface area of the test cell is 25 cm². If any dataset to be evaluated originated from a test cell area other than 25 cm², the impedance shall be multiplied by the total area then divided by 25 for each dataset.
- 8.2 The individual raw datasets shall be presented by Bode and Nyquist plots (6-8). The Bode plot is a Cartesian coordinate representation that provides the log impedance magnitude and phase angle versus log frequency. The Nyquist plot is a complex plane presentation that provides the negative imaginary impedance versus the real impedance. See Practices G3 and G106 for further information on these plots.
- 8.3 Compiled datasets may be presented as the impedance magnitude at 0.1 Hz, or otherwise agreed upon, to provide a graphical representation of the data and uniformity of coating quality across a structure.
- 8.3.1 *Scatter Plot*—Plot the impedance magnitude at 0.1 Hz, or otherwise agreed upon, versus location for structures that are linear or contain station referencing, such as a pipeline.

8.3.2 *Contour Map*—On a suitably scaled plan view of the structure surface, plot the impedance magnitude at 0.1 Hz, or otherwise agreed upon, for each test location and draw contours of equal impedance values through points of equal or interpolated equal values. The maximum contour interval shall be one order of magnitude.

Note 18—The impedance measurements are taken at a broad frequency range to ensure that a well-defined curve is produced, and the measurement error is low. More critical investigation of the low-frequency data provides for evaluation of the total resistance of the coating system. Complex structures can be illustrated using three-dimensional (3-D) plotting approaches if comprehensive impedance measurements are required and sections given by plotting on a plan view are not satisfactory. See Appendix X2 for information on interpretation of results.

9. Report

- 9.1 Report the following information:
- 9.1.1 Description of sampling plan, structure evaluated, location details, date of testing, and point of contact;
- 9.1.2 Measurement electrodes, whether a two- or threeelectrode setup was used, electrolyte solution, test cell hydration time, and potentiostat used;
- 9.1.3 Estimated environmental conditions, to include coating surface temperature and relative humidity;
- 9.1.4 Estimated coating condition, to include coating type, age, typical service conditions, and time since the coating surface became dry, if in immersion service;
- 9.1.5 The visual condition of each test area, noting any rust staining, pitting, abrasion, or similar defects in the area, and

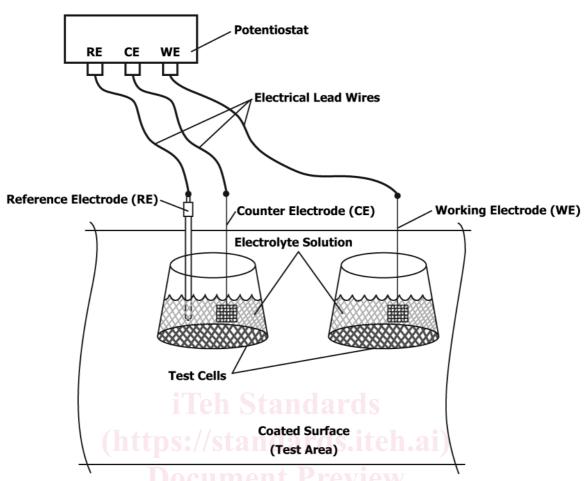


FIG. 2 Three-Electrode Measurement with No Electrical Connection to Substrate

test cell leakage if it occurs; test cell leakage is allowable provided that the measurement electrodes are submerged in electrolyte solution for the duration of the measurement and the leakage does not connect the two test cells across the coating surface;

- 9.1.6 The open circuit potential data taken before the impedance measurement and any observations regarding the stability of these data;
 - 9.1.7 Test cell surface area;
- 9.1.8 Nyquist and Bode plot for each location, unless otherwise agreed upon, noting whether the plots' scales are uniform or other;
- 9.1.9 Impedance magnitude at 0.1 Hz, unless otherwise agreed upon, versus location; clearly identify measurement frequency for the data plotted; and
- 9.1.10 The results of any validity measurements, if performed.

10. Precision and Bias

10.1 Precision—The single-laboratory repeatability variance has been determined to be 10 % using observed values of impedance magnitude at 0.1 Hz for five different coating materials (Table 1). The coatings were applied to steel plates and are typical of industrial or pipeline coatings used in water service. EIS measurements of the coated plates occurred

TABLE 1 Single-Laboratory Repeatability Results for Five
Different Coating Materials

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Coating Tested	Mean Mean Magnitude at 0.1 Hz (Ω)	Repeatability Variance, %	As 95 % Repeatability Upper Limit, Ω	95 % Repeatability Lower Limit, Ω
Material 1	3.9 × 10 ⁹	13.8	5.4 × 10 ⁹	2.4 × 10 ⁹
Material 2	3.8×10^{9}	6.7	4.5×10^{9}	3.1×10^{9}
Material 3	3.8×10^{9}	9.7	4.8×10^{9}	2.8×10^{9}
Material 4	1.2×10^{9}	20.1	1.8×10^{9}	5.2×10^{8}
Material 5	3.1×10^{8}	1.4	3.2×10^{8}	3.0×10^{8}
Mean		10.3		

outdoors using a portable potentiostat. The multiple-laboratory reproducibility will be developed within five years of approval of this standard.

10.2 *Bias*—When performed in accordance with this test method and accounting for the individual test areas and environmental conditions, there is no bias compared to a traditional laboratory EIS test.

11. Keywords

11.1 coating impedance; coating quality control; electrochemical impedance spectroscopy; EIS; field impedance; potentiostat