

Standard Practice for Immersion Testing of Industrial Protective Coatings and Linings¹

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1. Scope

- 1.1 This practice establishes procedures for the evaluation of the resistance of industrial protective coatings to immersion in chemicals.
- 1.2 Linings are a particular type of coating intended for protection of substrates from corrosion as a result of continuous or intermittent fluid immersion.
- 1.3 The values stated in SI units are to be regarded as the standard. The values given in parenthesis are for information only.
- 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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

C267 Test Methods for Chemical Resistance of Mortars, Grouts, and Monolithic Surfacings and Polymer Concretes

D16 Terminology for Paint, Related Coatings, Materials, and Applications

D471 Test Method for Rubber Property—Effect of Liquids D523 Test Method for Specular Gloss

D610 Practice for Evaluating Degree of Rusting on Painted Steel Surfaces

D714 Test Method for Evaluating Degree of Blistering of Paints

D785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

- D1474 Test Methods for Indentation Hardness of Organic Coatings
- D1734 Practice for Making Cementitious Panels for Testing Coatings
- D2200 Practice for Use of Pictorial Surface Preparation Standards and Guides for Painting Steel Surfaces
- D2240 Test Method for Rubber Property—Durometer Hardness
- D2583 Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor
- D3359 Test Methods for Measuring Adhesion by Tape Test
 D3363 Test Method for Film Hardness by Pencil Test
- D4138 Practices for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive, Cross-Sectioning Means
- D4417 Test Methods for Field Measurement of Surface Profile of Blast Cleaned Steel
- D4538 Terminology Relating to Protective Coating and Lining Work for Power Generation Facilities
- D4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers
- D5139 Specification for Sample Preparation for Qualification Testing of Coatings to be Used in Nuclear Power Plants
- D5162 Practice for Discontinuity (Holiday) Testing of Nonconductive Protective Coating on Metallic Substrates
- D6132 Test Method for Nondestructive Measurement of Dry Film Thickness of Applied Organic Coatings Using an Ultrasonic Coating Thickness Gage
- D6577 Guide for Testing Industrial Protective Coatings
- D6677 Test Method for Evaluating Adhesion by Knife
- D7055 Practice for Preparation (by Abrasive Blast Cleaning) of Hot-Rolled Carbon Steel Panels for Testing of Coatings
- D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals
- D7234 Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers

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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.



G113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials

2.2 NACE Standard:³

TM 0174-2002 Laboratory Methods for the Evaluation of Protective Coatings and Lining Materials on Metallic Substrates in Immersion Service

2.3 SSPC/NACE Joint Standards:⁴

SSPC-SP5/NACE 1 White Metal Blast Cleaning

SSPC-SP10/NACE 2 Near White Blast Cleaning

SSPC-SP13/NACE 6 Surface Preparation of Concrete

SSPC Protective Coatings Glossary

2.4 Other Document:⁵

Coating Encyclopedia Dictionary

3. Terminology

3.1 The definitions given in Terminologies D16, G113 and D4538 are applicable to this practice.

4. Significance and Use

- 4.1 Protective coatings are used on metallic and concrete storage and processing vessels, shipping containers, dams and rail cars to protect the substrate from corrosive attack and to protect stored materials (cargo) from contamination. This method provides a means to assess the ability of a protective coating to resist degradation by chemicals and to protect the liquid cargo from contamination by either the substrate or coating, based on visual observations. Other measures of degradation, such as changes in weight or dimensions of the coating material, or chemical changes to the cargo, may be used to assess this protective ability as mutually agreed upon between contracting parties. Simple chemical-resistance evaluations of the lining materials may be performed more conveniently by other pertinent methods as a prescreening test for this procedure in accordance with Test Methods C267 and D471.
- 4.2 This practice covers three approaches to conducting evaluations of a lining coating material's fitness for purpose.
- 4.2.1 *Method A*—Evaluation of specimens under conditions of constant temperature at atmospheric pressure, (that is, without a thermal gradient).
- 4.2.2 *Method B*—Evaluation of specimens under conditions which provide a temperature gradient across the sample.
- 4.2.3 *Method C*—Evaluation of specimens under conditions of constant temperature and increased pressure, (that is, without a thermal gradient).

5. Preparation of Test Specimens

- 5.1 Metallic Panels:
- 5.1.1 Use carbon steel panels with a minimum test size of 50 by 100 mm (2 by 4 in.), minimum thickness 3 mm (1/8 in.), unless otherwise agreed upon by purchaser and supplier.
- ³ Available from NACE International (NACE), 1440 South Creek Dr., Houston, TX 77084-4906, http://www.nace.org.
- ⁴ Available from Society for Protective Coatings (SSPC), 40 24th St., 6th Floor, Pittsburgh, PA 15222-4656, http://www.sspc.org.
- ⁵ Available from Federation of Societies for Coatings Technology (FSCT), 492 Norristown Rd., Blue Bell, PA 19422-2350, http://www.coatingstech.org.

- Note 1—Test plates with a minimum width of 25 mm (1 in.) may be used if the film thickness is sufficiently uniform to prevent edge effects.
- 5.1.2 Use nominal 4.8 mm (3/16 in.) thick carbon steel panels in coating tests if a temperature gradient is included in the immersion test, unless otherwise agreed upon by purchaser and supplier.
- 5.1.3 The minimum surface preparation is abrasive blast cleaning to Near-White Metal condition as defined by Practice D2200 and by SSPC-SP10/NACE 2. The abrasive and feed pressure shall be selected to produce an anchor pattern, which is compatible with the coating system and acceptable to the coating manufacturer.
- 5.1.4 Unless otherwise specified or agreed upon by purchaser and seller, surface preparation shall be "White Metal Blast Cleaning" according to SSPC-SP5/NACE 1, with surface profile of 35 to 90 micrometers (1.5 to 3.5 mils) as determined by Method C of Test Methods D4417. If the product recommendation falls outside of the range of Test Methods D4417 then prepare the test panels in accordance with Practice D7055.
- 5.1.5 Other metallic substrates, panel dimensions, or surface preparation are acceptable if agreeable to concerned parties.
 - 5.2 Concrete and Cementitious Panels:
- 5.2.1 *Cementitious Panels*—The minimum panel size is 50 by 100 by 12 mm (2 by 4 by ½ in.). Prepare cementitious panels by abrasive blast cleaning in accordance with SSPC-SP13/NACE 6 or by other method agreed upon between purchaser and supplier.
- 5.2.2 Concrete Blocks—Form, condition and prepare concrete blocks in accordance with the protocol described in Specifications D5139 or D1734. Prepare concrete blocks by abrasive blast cleaning in accordance with SSPC-SP13/NACE 6 or by other method agreed upon between purchaser and supplier.
 - 5.3 Coating Application:
- 5.3.1 For panels to be fully immersed, apply the coating system to all test panel surfaces that will be subjected to exposure.
- 5.3.2 Apply the coating system according to the manufacturer's instructions appropriate for actual or anticipated service conditions. Edges should be protected unless the intent of the experiment is to evaluate the performance of the coating on edges. For coatings to be tested under Method A (immersion with no temperature gradient) or Method C (immersion at high pressure), apply the candidate coating to the back of the panel in addition to the front and the edges to prevent effects from dissimilar coatings.
- 5.3.3 Dry film thickness of the coating shall be agreed upon between purchaser and seller.
- 5.3.3.1 Measure dry film thickness of coating applied to metal surfaces in accordance with Practice D7091.
- 5.3.3.2 Measure dry film thickness of linings applied to concrete or cementitious surfaces in accordance with Test Method D6132. Alternative 1, measure dry film thickness of linings applied to concrete or cementitious surfaces by using sacrificial surfaces prepared concurrently with test specimens in accordance with Practices D4138. A second alternative is to

simultaneously prepare a steel panel, placed next to one of the concrete panels, and then measure its DFT in accordance with Practice D7091.

5.3.4 Examine the coated surface for holidays in accordance with Practice D5162. For test plates and concrete blocks, no holidays are acceptable unless agreed upon between purchaser and seller.

Note 2—High-voltage holiday detection should not be used on linings that have been exposed. The test could be destructive and may not be meaningful since the dielectric strength of the lining material may be changed by the exposure.

- 5.3.5 Visually inspect the lining surface of all panels before test exposure is begun and note any gross imperfections such as voids, cracks, runs or sags.
- 5.3.6 If hardness is required determine before exposure by using a suitable standard such as Test Methods D785, D1474, D2240, D2583, or D3363 on an unexposed reference panel or in the case of an atlas cell in an area of the panel that will not be exposed to the test solution.

6. Sampling

- 6.1 Test chemicals are to be representative of test materials expected in actual service.
- 6.2 The concentration(s) of the chemical(s) shall be specified. Unless otherwise stated, all dilutions shall be made with distilled, demineralized or deionized water.
- 6.3 If no concentration is specified, it is understood that the chemical is used in its undiluted form.
- 6.4 Prepare at least two test panels or blocks per coating system for each test material.
- 6.5 Prepare at least one test panel or block per coating system to be used as an unexposed reference (file specimen).
- 6.6 Exposure of materials of known performance (a control) at the same time as the test material is recommended.
- 6.7 Retain an unused portion of each test medium as a blank if analysis of the test solution is performed.

7. Test Methods

Note 3—This standard describes three methods for immersion testing of protective coatings and linings. Section 8 provides recommended examination methods and intervals between evaluations for the methods described below.

- 7.1 Method A—Immersion with No Temperature Gradient:
- 7.1.1 Test Apparatus:
- 7.1.1.1 A closed vessel constructed of glass or other inert material is used with capacity to hold one or more test specimens with means to relieve pressure if operated at elevated temperatures.
 - Note 4—Slowly evaporating media may not require a closed vessel.
- 7.1.1.2 Specimens should be positioned so as to avoid specimen to specimen contact.
- 7.1.1.3 For immersion at high or low temperature, insulation of the vessel is recommended. A separate test vessel is recommended for each type of coating system being evaluated. For elevated temperature immersion, an apparatus as described in NACE TM 0174-2002, Procedure B, is recommended.

7.1.2 *Test Medium*—The test solution is selected to correspond with the anticipated service of the coating. Add enough of the fluid to immerse ½ to ¾ of the test panel. The fluid level should be checked at least once every 7 days. If more than 5 % of the test fluid evaporates from the test vessel, add enough fluid to maintain the level and composition of the fluid.

Note 5—When solvent or water evaporates, ensure that the addition results in the same concentration as at the start of the test.

- 7.1.3 *Test Procedure*—Maintain a temperature of $25 \pm 2^{\circ}$ C (77 $\pm 4^{\circ}$ F) unless otherwise indicated by anticipated service conditions or by specification. Ensure that there is a minimum separation of 25 mm (1 in.) between panels in the vessel.
- 7.2 Method B—Immersion with Temperature Gradient—The practice defines 2 types of temperature gradients: increasing temperature from uncoated to coated side and decreasing temperature from uncoated to coated side.

Note 6—The specific equipment drawings and operation are described in NACE TM 0174-2002, Procedure A.7.2.1

- 7.2.1 *Method B1*—Temperature gradient with temperature increasing from uncoated to coated.
- 7.2.1.1 *Test Apparatus*—A special cylindrical glass test cell is used with multiple necks to provide inlets for thermometer, heater and condenser. The test panel is coated on one side with the exterior exposed to the ambient environment. This setup provides a temperature gradient across the coating.
- 7.2.1.2 *Test Medium*—The test solution is selected to correspond with the anticipated service of the coating. As this is a closed system there is no need to replenish the solution during the time test specimen is immersed. After disassembly and rating of the panel, fresh medium is normally recommended.
- 7.2.1.3 *Test Procedure*—Clamp the test panels in place at the ends of the test cell with the coating positioned against the cell interior. Fill the test cell to $\frac{2}{3}$ to $\frac{3}{4}$ of its total height with the test solution and commence heating.

Note 7—If temperature is not specified then the default conditions for an increasing temperature gradient should be $23\pm2^{\circ}C$ ($73.4\pm3.6^{\circ}F$) external and $60\pm2^{\circ}C$ ($140\pm3.6^{\circ}F$) internal cell temperature.

- 7.2.2 *Method B2*—Temperature gradient with temperature decreasing from uncoated to coated.
- 7.2.2.1 *Test Apparatus*—Use a vessel as described in 7.2.1.1 but with heating element attached to the uncoated exterior panel; or install panel with the uncoated side on the hot plate and the chemical container filled with chemical to the coated side
- 7.2.2.2 *Periodic Cycling*—Use periodic cycling (heating/cooling) as requested.
- 7.2.2.3 Test Medium—The test solution is selected to correspond with the anticipated service of the coating. As this is a closed system there is no need to replenish the solution during the time test specimen is immersed. After disassembly and rating of the panel, fresh medium is normally recommended.
- 7.2.2.4 *Test Procedure*—Clamp the test panels in place at the ends of the test cell with the coating positioned against the cell interior. Fill the test cell to ½3 to ¾ of its total height with the test solution and commence heating.

- Note 8—If temperature is not specified then the default conditions for a decreasing temperature gradient should be $60 \pm 2^{\circ}\text{C}$ ($140 \pm 3.6^{\circ}\text{F}$) external and $23 \pm 2^{\circ}\text{C}$ ($73.4 \pm 3.6^{\circ}\text{F}$) internal cell temperature.
- 7.3 Method C—Immersion Testing at Static Temperature and Elevated Pressure:
- 7.3.1 *Test Apparatus*—The test apparatus is an insulated autoclave having an external heat source and a capability of being operated at the temperatures and pressures agreed upon by purchaser and supplier.
- 7.3.2 *Test Conditions*—The test medium and pressure are selected to correspond with the anticipated service of the coating.
 - 7.3.3 Test Procedure:
- 7.3.3.1 Immerse the test panels $\frac{2}{3}$ to $\frac{3}{4}$ in the test fluid, or as otherwise specified between the purchaser and supplier.
- 7.3.3.2 If the external heat source is needed, then raise the temperature as agreed upon between the purchaser and supplier.
- 7.3.3.3 Raise the pressure of the autoclave as specified between the purchaser and supplier.
 - 7.3.4 Periodic Cycling:
- 7.3.4.1 To allow initiation of discharging or recharging of a vessel, the test panels are removed from the autoclave for a two-day period once every two weeks. Panels are allowed to reach room temperature at this time.
- 7.3.4.2 Test panels are returned to the immersion apparatus after the cycling ends. The test apparatus is restored to the conditions described in 7.3.3 and 7.3.4.

Note 9—If temperature and/or pressure is not specified then the default conditions should be 43 \pm 2°C (109.4 \pm 3.6°F) in the vessel and 3 bar (43.5 psi) in the autoclave.

8. Examination and Evaluation

- 8.1 Examination Schedule:
- 8.1.1 For the exposures described in 7.1 (Method A, immersion with no temperature gradient) and 7.2 (Methods B1 and B2, immersion with temperature gradient), the inspection schedule shall be agreed upon between purchaser and seller.

Note 10—It is suggested to visually inspect the test panels after one week in immersion, the second evaluation at one month, and monthly thereafter.

- 8.1.2 For the exposures described in 7.3 (Method C, immersion at elevated temperature and pressures), inspect test panels at the completion of each cycling period.
 - 8.2 Duration of Test:
- 8.2.1 Run the immersion test for a minimum of one month, or other time as agreed to by purchaser and supplier.
- 8.2.2 Alternately run the immersion until a predetermined level of failure is reached. Predefined failure may be based on a purchaser or performance specification or coating manufacturer's internal standards.
 - 8.3 Examination Procedures:

- 8.3.1 At all inspections, visually evaluate the test panels on portions of the panel exposed to liquid solution, vapor phase, liquid-vapor interface, and compare to an unexposed area or reference panel. Record the results for blistering (Test Method D714), rusting (Test Method D610), lifting, peeling, discoloration, erosion, softening, etching, wrinkling, cracking, swelling, dissolving, delamination, and changes in gloss relative to the file test panel (Test Method D523, and any other changes observed. Other non-destructive tests such as weight change or thickness change as agreed to by purchaser and seller may also be performed. Make separate ratings for coating areas exposed to the test liquid and its vapor phase, where both services are tested.
- 8.3.2 Additional suggested test procedures include Test Method D3363 and D2240 for hardness, and Test Methods D3359, D6677, D7234 and D4541 for adhesion. Compare the results of the exposed panel to the results of the unexposed reference. These tests are often only performed at the conclusion of the test as they are destructive. These tests (except for Test Method D4541) should be conducted within 30 min of the removal of the coated specimens from the immersion. It may be desirable to reevaluate the coating system after a specified recovery time. Other tests as described in Guide D6577 may also be performed as agreed between purchaser and supplier.
- 8.3.3 At all inspections of the exposures described above, visually evaluate changes in the test medium relative to the retained exposure medium. Record changes observed in color or turbidity of the immersion medium and the presence of any precipitates or changes in pH where applicable.
- 8.3.4 High-voltage holiday detection should not be used on linings that have been exposed. The test could be destructive and may not be meaningful since the dielectric strength of the lining material may be changed by the exposure.

9. Documentation and Report

- 9.1 Report the following information for each coating system evaluated.
 - 9.1.1 Identification of the coating system tested,
- 9.1.2 Details of type of substrate surface preparation, coating application, and any application defects,
 - 9.1.3 Film thickness of each coating layer,
 - 9.1.4 Curing schedule,
- 9.1.5 Chemical identity of the test materials and test conditions,
 - 9.1.6 Observations made at each inspection,
- 9.1.7 Failure criteria if "time to failure" was used to assess coating resistance to test exposure,
 - 9.1.8 Recovery time if applicable, and
 - 9.1.9 Specific test procedure

10. Keywords

10.1 chemical; concrete; cycling; evaluation; immersion; panels; steel; temperature gradient; testing

APPENDIX

(Nonmandatory Information)

X1. CHEMICAL IMMERSION SAMPLE RESULTS SHEET

X1.1 See Fig. X1.1.

Sample

Nature of EffectBlisteringBCrackingCDiscolorationDcDissolvingDsLiftingLfLoss of GlossLGNo EffectNeRustingRSofteningSfSwellingSwWrinklingW	ID:Start: Cure: Chemical:		_	Project: Substrate DFT:		
	Blistering Cracking Discoloration Dissolving Lifting Loss of Gloss No Effect Rusting Softening Swelling	B C Dc Ds Lf LG Ne R Sf Sw	Extent	Very Slight Slight Moderate	S M	

Exposure Time Interval

Product						

FIG. X1.1 Chemical Immersion Sample Results Sheet



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