



Designation: D6943 – 10

Standard Practice for Immersion Testing of Industrial Protective Coatings¹

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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 The values stated in SI units are to be regarded as the standard. The values given in parenthesis are for information only.

1.3 *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

- C868 Test Method for Chemical Resistance of Protective Linings
- D16 Terminology for Paint, Related Coatings, Materials, and Applications
- D610 Practice for Evaluating Degree of Rusting on Painted Steel Surfaces
- D714 Test Method for Evaluating Degree of Blistering of Paints
- D2200 Practice for Use of Pictorial Surface Preparation Standards and Guides for Painting Steel Surfaces
- 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
 - 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 Document⁵
- Coating Encyclopedia Dictionary

¹ This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

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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.

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>.

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.

4.2 This standard practice covers three approaches to conducting evaluations of a lining coating materials 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 provides a temperature gradient across the sample: The temperature may increase from coated side to uncoated side (Method B1) or decrease from coated side to uncoated side (Method B2).

4.2.3 *Method C*—Evaluation of specimens under conditions of constant temperature and increased pressure, (that is, without a thermal gradient).

4.3 Exposure of materials of known performance (a control) at the same time as the test material is recommended.

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.

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 recom-

mendation 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 1/2 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 Specification **D5139**. 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**. Alternatively, 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**.

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.

6. Sampling

6.1 Test chemicals are to be representative of test materials expected in actual service.

6.2 Prepare at least two test panels or blocks per coating system for each test material.

6.3 Prepare at least one test panel or block per coating system to be used as an unexposed reference (file specimen).

6.4 Retain an unused portion of each test medium as a blank.

7. Test Methods

NOTE 2—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 3—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 one half to two thirds 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 4—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 $25 \pm 2^\circ\text{C}$ ($77 \pm 4^\circ\text{F}$) 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

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.

NOTE 5—The specific equipment drawings and operation are described in Test Method C868 and NACE TM 0174-2002, Procedure A.

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.

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 $\frac{2}{3}$ to $\frac{3}{4}$ of its total height with the test solution and commence heating.

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 in the vessel to $43 \pm 2^\circ\text{C}$ ($110 \pm 4^\circ\text{F}$), or as otherwise specified between the purchaser and supplier.

7.3.3.3 Raise the pressure of the autoclave to 3 bar (45 psi) or as otherwise 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.

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 6—It is suggested to visually inspect the test panels daily for the first few days to identify any catastrophic failures, weekly for the first 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: