Designation: D6944 – 15

Standard Practice for Determining the Resistance of Cured Coatings to Thermal Cycling[1]

This standard is issued under the fixed designation D6944; 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 practice determines the resistance of cured coatings or coating systems to repeated thermal cycles and is designed to assess the effect of thermal cycling on the properties of a coating or coating system. These properties may include adhesion, resistance to checking, cracking, blistering, or others. This procedure is not intended to provide a quantitative measure of the service life that can be expected from a specific coating system on a given substrate.

1.2 *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]

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

D3359 Test Methods for Measuring Adhesion by Tape Test

D4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers

D6132 Test Method for Nondestructive Measurement of Dry Film Thickness of Applied Organic Coatings Using an Ultrasonic Coating Thickness Gage

D6677 Test Method for Evaluating Adhesion by Knife

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

G147 Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests

3. Summary of Test Methods

3.1 *Test Method A*—Cured test specimens are subjected to 30 thermal cycles of immersion, freezing and heating.

3.2 *Test Method B*—Cured test specimens are subjected to 30 thermal cycles of freezing and heating.

4. Significance and Use

4.1 The purpose of this test is to obtain information on the ability of a coating system to adhere to substrates under thermal stress. It is assumed that the coating systems tested are applied and cured according to the coating manufacturer's instructions.

TEST METHOD A—FREEZE/THAW/IMMERSION

5. Apparatus

5.1 *Thermal Cycling Apparatus:*

5.1.1 The thermal cycling apparatus shall consist of a suitable chamber or chambers in which the test specimens may undergo the specified cycle. Ideally, a chamber which contains heating and refrigeration equipment and immersion capabilities in the same unit should be used. This chamber shall have the ability to maintain continuous reproducible cycles within the specified temperature requirements. In the event that an apparatus having freezing, heating, and immersion capabilities is not available, separate equipment for freezing, heating and immersion may be used.

5.1.2 The chamber or chambers shall have the ability to maintain a constant temperature during each of the respective temperature intervals as specified by the procedure.

5.1.3 The samples shall be arranged to minimize contact with the chamber surfaces or any mounting racks, and to maximize air flow.

[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.

5.1.4 The temperature of the thermal cycling apparatus shall give uniform readings at various locations within the chamber, within 3°C (5°F) at any given time, except during the transition between heating and freezing cycles. A two hour temperature ramping period is permitted for the equipment to reach the next temperature setting.

6. Test Specimens

6.1 Apply each coating or coating system onto the substrate type and thickness as agreed upon. Preparation of the substrate (that is, surface cleanliness and roughness) prior to application of the coating or coating system shall be agreed upon. Measure the thickness of each coating layer in accordance with Practice D7091 (metallic substrates) or Test Method D6132 for non-metallic substrates.

6.2 Apply each coating or coating system to a minimum of two specimens to determine repeatability. If the substrate of choice is concrete, then suitable encapsulation of the substrate must occur as agreed upon between purchaser and supplier.

6.2.1 When destructive tests (for example, adhesion) are required, a sufficient number of reference specimens shall be retained so that the property of interest can be determined on non-cycled reference specimens each time cycled test specimens are evaluated.

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

6.3 Follow the procedures described in Practice G147 for identification, conditioning and handling of specimens of test, control, and reference materials prior to, during, and after cycling.

6.4 Unless otherwise specified, cure each test specimen for a minimum of 7 days at 23 ± 3°C (73 ± 5°F) before beginning thermal cycling.

6.5 Prior to beginning the thermal cycling, determine relevant properties of the coating on a reference panel. Test Methods D660, D661, D714, D3359, and/or D4541, and/or D6677 and/or D7234 are recommended. Consider the intended service environment and product use requirements when selecting appropriate evaluation methods.

7. Procedure

7.1 After the curing period, place the specimens into the thermal cycling apparatus or test chambers and begin the cycling procedure.

7.1.1 A suitable procedure is shown in the table below. Other procedures/temperatures/immersion media as agreed upon between purchaser and supplier may also be used.

7.1.2 *Thermal Cycling Procedure:*

Condition	Time (h)
50 ± 3°C (122 ± 5°F) in air	4
25 ± 3°C (77 ± 5°F) tap water immersion	4
-29 ± 3°C (–20 ± 5°F) in air	16

7.2 Repeat this cycle of heat/immersion/freeze for 30 cycles, or other agreed upon number of cycles. Leave the surface of the panels wet when transferring from the immersion to the freeze cycle. Maintain panels in the freeze condition upon interruptions in the cycling (for example, weekends/holidays). Evaluate specimens either every 5 cycles or at the end of 30 cycles. Destructive tests are typically performed at the completion of all cycles.

TEST METHOD B—FREEZE/THAW

8. Apparatus

8.1 *Thermal Cycling Apparatus:*

8.1.1 The thermal cycling apparatus shall consist of a suitable chamber or chambers in which the test specimens may undergo the specified cycle. Ideally, a chamber which contains heating and refrigeration in the same unit should be used. This chamber shall have the ability to maintain continuous reproducible cycles within the specified temperature requirements. In the event that an apparatus having freezing and heating capabilities is not available, separate equipment, one chamber for freezing and another for heating may be used.

8.1.2 The chamber or chambers shall have the ability to maintain a constant temperature during each of the respective temperature intervals as specified by the procedure.

8.1.3 The samples shall be arranged to minimize contact with the chamber surfaces or any mounting racks, and to maximize air flow.

8.1.4 The temperature of the thermal cycling apparatus shall give uniform readings at various locations within the chamber, within 3°C (5°F) at any given time, except during the transition between freezing and heating. A two hour temperature ramping period is permitted for the equipment to reach the next temperature setting.

9. Test Specimens

9.1 Test specimens shall be as described in Section 6.

10. Procedure

10.1 After the curing period, place the specimens into the thermal cycling apparatus or test chambers and begin the cycling procedure.

10.1.1 A suitable procedure is shown in the table below. Other procedures/temperatures as agreed upon between purchaser and supplier may also be used.

10.1.2 *Thermal Cycling Procedure:*

Condition	Time (h)
50 ± 3°C (122 ± 5°F) in air	8
-29 ± 3°C (-20 ± 5°F) in air	16

10.2 Repeat this cycle of heat/freeze for 30 cycles, or other agreed upon number of cycles. Maintain panels in the freeze condition upon interruptions in the cycling (for example, weekends/holidays). Evaluate specimens either every 5 cycles or at the end of 30 cycles. Destructive tests are typically performed at the completion of all cycles.

11. Report

11.1 Test Method used and number of testing cycle(s).

11.2 Cycle temperatures.

11.3 Results of the visual evaluation of each coated panel for the degree of checking as described in Test Method D660 (if used).

11.4 Results of the visual evaluation of each coated panel for the degree of cracking as described in Test Method D661 (if used).

11.5 Results of the visual evaluation of each coated panel for the degree of blistering as described in Test Method D714 (if used).

11.6 Results of adhesion testing from one or more of Test Methods D3359, D4541, D6677, and/or D7234 (if used), compared to the adhesion properties of non-cycled coated panels.

11.7 Description of test specimens, including substrate type and thickness, surface preparation (degree of cleanliness and roughness), coating type and thickness and cure time/conditions prior to cycling.

12. Keywords

12.1 freeze/thaw; freezing; heating; immersion; thawing; thermal cycling; thermal stress