



Designation: D 6944 – 03

Standard Test Method for Resistance of Cured Coatings to Thermal Cycling¹

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1. Scope

1.1 This test method determines the resistance of cured coatings to repeated thermal cycles. This procedure is designed to assess the influence of thermal cycling on adhesion and other properties of coatings. 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:

- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers³
- D 6677 Test Method for Evaluating Adhesion by Knife³
- G 147 Practice of Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests⁴

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 used are applied and cured according to the given specifications of the coating manufacturer.

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.

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 onto the substrate as agreed upon. Minimum film thicknesses should take substrate profile into account when applied.

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 D01.46 on Industrial Protective Coatings.

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² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 14.04.

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 are run, it is recommended that a sufficient number of file specimens be retained so that the property of interest can be determined on unexposed file specimens each time exposed materials are evaluated.

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

6.3 Follow the procedures described in Practice **G 147** for identification, conditioning and handling of specimens of test, control, and reference materials prior to, during, and after exposure.

6.4 Unless otherwise specified, cure each test specimen for a minimum of 7 days at $23 \pm 3^\circ\text{C}$ ($73 \pm 5^\circ\text{F}$) before beginning thermal cycling.

6.5 Prior to beginning the thermal cycling, determine relevant properties of the coating on a reference panel. Test Methods **D 660**, **D 661**, **D 714**, **D 3359**, and/or **D 4541**, and/or **D 6677** are recommended. Consider product use requirements when selecting appropriate methods.

7. Procedure

7.1 After the curing period, place the specimens into the test chamber and begin the cycling procedure.

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

7.1.2 Thermal Cycling Procedure:

Condition	Time (h)
$-5 \pm 3^\circ\text{C}$ ($23 \pm 5^\circ\text{F}$) in air	16
$50 \pm 3^\circ\text{C}$ ($122 \pm 5^\circ\text{F}$) in air	4
$25 \pm 3^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$) tap water immersion	4

7.2 Repeat this cycle of freeze/thaw/immersion 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. Keep panels in the freeze condition upon interruptions in the agreed upon cycle. Evaluate specimens either every 5 cycles or at the end of 30 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.

8.2 Reproducibility of test results between laboratories has been shown to be good when the stability of materials is evaluated in terms of performance ranking compared to other materials or to a control. Therefore, exposure of a similar material of known performance (a control) at the same time as the test materials is strongly recommended.

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 test chamber and begin the cycling procedure.

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

10.1.2 Thermal Cycling Procedure:

Condition	Time (h)
$-5 \pm 3^\circ\text{C}$ ($23 \pm 5^\circ\text{F}$) in air	16
$50 \pm 3^\circ\text{C}$ ($122 \pm 5^\circ\text{F}$) in air	8

10.2 Repeat this cycle of freeze/thaw for 30 cycles, or other agreed upon number of cycles. Keep panels in the freeze condition upon interruptions in the agreed upon cycle. Evaluate specimens either every 5 cycles or at the end of 30 cycles.

11. Report

11.1 Test Method used and testing cycle(s).

11.2 Visual evaluation of each panel for the degree of checking of exterior paints as described in Test Method **D 660**.

11.3 Visual evaluation of each panel for the degree of cracking of exterior paints as described in Test Method **D 661**.

11.4 Visual evaluation of each panel for the degree of blistering of exterior paints as described in Test Method **D 714**.

11.5 Results of adhesion testing from one or more of Test Methods **D 3359**, **D 4541**, and **D 6677**.

11.6 Identification of test specimens, including substrate, surface preparation, type and thickness of coating, and cure conditions.

12. Precision and Bias

12.1 The precision and bias are dependent upon the specific evaluations performed after the test cycles for each given coating system and is therefore beyond the scope of this test method.

13. Keywords

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