Designation: D2243 – 20

Standard Test Method for Freeze-Thaw Resistance of Water-Borne Coatings[1]

This standard is issued under the fixed designation D2243; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers a procedure for evaluating the effect of freeze/thaw cycling on the viscosity and visual film properties of water-borne coatings.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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

2.1 *ASTM Standards:*[2]

D523 Test Method for Specular Gloss

D562 Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer

D714 Test Method for Evaluating Degree of Blistering of Paints

D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer

D2244 Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

D2805 Test Method for Hiding Power of Paints by Reflectometry

3. Summary of Test Method

3.1 The water-borne coating is put into at least two pint-size (500-mL) resin-lined cans. One can is stored at room temperature, while the other cans are subjected to cycles of freezing and thawing. After cycling, the paint samples are examined for changes in viscosity and visual film properties.

3.2 It is critical that the test cans be undisturbed until they reach the number of freeze-thaw cycles at which they are to be tested. This condition simulates actual freeze-thaw behavior of paints in the field.

4. Significance and Use

4.1 When water-borne coatings are shipped during cold weather, they may experience cycles of freezing and thawing. Cycles of freezing and thawing cause more damage to water-borne coatings than when the coatings are subjected to steady freezing.

5. Apparatus

5.1 *Test Chamber*—A suitable cabinet, room, or enclosure space large enough to contain the specimens to be tested permitting at least 25 mm (1 in.) of air space between the sides of adjacent cans and capable of being maintained continuously at a temperature of −18°C (0°F).

NOTE 1—Although a variation of the test chamber temperature of ±2°C (±3.5°F) is allowed, the test chamber temperature should be maintained as near −18°C (0°F) as practicable and the amount of variation should be recorded and reported.

5.2 *Viscometer*—A Stormer viscometer with paddle type rotor as described in Test Method D562 or a Brookfield viscometer as described in Test Methods D2196.

5.3 *Test Charts*—Smooth surface paper charts having adjacent black and white areas, and coated with a suitable varnish or lacquer to render the surface impermeable to paint liquids.

5.4 *Paint Brush,* 25 mm (1 in.).

5.5 *Bar Applicator,* with a 0.18-mm (7-mil) clearance.

[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 D01.42 on Architectural 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.

6. Preparation of Sample and Specimens

6.1 Prepare specimens for testing by filling 500 mL (1-pt) resin-lined, friction-top cans two thirds full. Ensure that the bulk sample from which the cans are filled is well stirred and uniform, that the containers used are clean, and that the lids are applied promptly to the cans to prevent evaporation losses. At least two such specimens are required for each test.

6.1.1 When working with limited quantities of paint, the use of 250 mL (½-pt) cans is acceptable, although may be contrary to the intent of the method. Following this modified use may result in more severe results that may not be seen with the larger container.

6.2 At least two such specimens are required for each test, the control (unfrozen) sample and the final test sample. If it is the intent of the test to check where a sample fails between one and five freeze/thaw cycles, a sample for each of the cycles tested should be made. For clarity, each of the samples should also be labeled corresponding to the number of cycles for which that specimen should be tested.

Example: If testing for each of five freeze/thaw cycles, a total of six cans will be required (one room temperature control, five for freeze/thaw testing, where one can will only get one freeze/thaw cycle, one will get two cycles, etc.). See Appendix X1.

6.2.1 Using only one container for freeze/thaw testing, and opening the can between cycles, mixing to check for failure, then placing the container back in for further cyclic testing is not proper procedure for this method. Disturbing the paint by mixing between cycles will not represent the field failures that go undisturbed between cycles.

7. Exposure to Test Conditions

7.1 Store one can at room temperature and identify this as the control specimen.

7.2 Place properly labeled additional cans of paint being tested (identified as “test specimen – 1 cycle,” “test specimen – 2 cycles,” “test specimen – 3 cycles,” etc. as needed) in the chamber maintained at −18°C (0°F) in such a manner that they do not touch the walls or bottom of the chamber and so that free circulation of air around them is permitted. The placing of cans on racks that raise them off the bottom of the chamber or upon pieces of insulating board resting on the bottom is suggested. In the case of several test specimens, maintain a minimum of 25 mm (1 in.) of air space between adjacent cans and between cans and the chamber walls. Keep the test specimens in the chamber for 17 h and then remove and allow to stand for 7 h undisturbed at room temperature, adjacent to the control specimen, for a complete freeze-thaw cycle of 24 h.

7.3 Repeat 7.2 for additional freeze-thaw cycles, as many as agreed on between cooperating laboratories or buyer and seller (One to five cycles are usual.).

7.4 For testing that requires a bit more differentiation for low-VOC paints, Method B is similar to the original Method A, except instead of allowing the cans to sit undisturbed for 7 h at room temperature, an additional 24 h of thaw (for a total of 31 h of thaw) is used, making the complete freeze-thaw cycle of 48 h.

8. Examination

8.1 After completion of the agreed or specified number of cycles, examine both the test and control specimens for condition in the can, rating any evidence of settling, gelation, coagulation as follows:

10 = none	Good consistency paint that can be measured on a Stormer Viscometer.
8 = very slight	Forms a thick (or thin), flowable paint that cannot be read on a Stormer Viscometer. Also may have settled into a thick layer and a clear layer (settled out), that can be mixed back together, but still cannot be read on Stormer Viscometer.
6 = slight	Forms a thick mass, with a dough-like consistency.
4 = moderate	Forms chunks, like thick cottage cheese.
2 = considerable	Solidifies hard and rubbery, like semi-pliable putty.
0 = complete failure	Solidifies hard, like a hockey puck. May actually settle into a hard layer and a clear fluid layer.

8.2 Stir the test and control paint by hand and measure their viscosities (if possible) in accordance with Test Methods D562 or D2196. Record temperature of measurement.

NOTE 2—Stir specimens by hand in their can using a spatula. Stir carefully so as to avoid air entrainment and foam.

NOTE 3—In carrying out consistency determinations using Test Method D562, specimens should be maintained at 23 ± 2°C (73.5 ± 3.5°F) until two successive readings agree within 1 KU. As is the case with all non-Newtonian fluids, viscosity variation is dependent to a degree upon intensity and duration of agitation. Take utmost care that control and test specimens receive identical treatment during all stirring operations.

8.3 Immediately following viscosity determinations, apply both the control and test coating to a test panel (5.3) using the 0.18-mm (7-mil) clearance blade applicator (5.4). Allow to dry for at least 24 h and then visually compare the test versus the control coating for changes in speckiness, agglomeration, or coagulation. Rate changes as follows, or (if agreed upon) as indicated using Test Method D714 pictorial standards:

10 = none	4 = moderate
8 = very slight	2 = considerable
6 = slight	0 = complete failure

8.4 Do instrumental comparisons of the test versus the control coating for gloss (Test Method D523), hiding (Test Method D2805), and color change (Practice D2244). Also, report difference in viscosity (KU) if possible.

9. Report

9.1 Report the following information:

9.1.1 Report the condition of the paint in the can, in accordance with 8.1,

9.1.2 Report the change in viscosity between the test and control paints (KU or Brookfield, or both), in accordance with 8.2,

9.1.3 Report the rating of any visual differences between the test and control paints, in accordance with 8.3,

9.1.4 Report the rating of any instrumental differences between the test and control paints, in accordance with 8.4, and

9.1.5 Report whether the testing consisted of the 24-h cycle test or the 48-h cycle test.

10. Precision and Bias

10.1 *Precision*—The precision of this test method in regard to viscosities is as specified in Test Methods D562 or D2196, whichever is employed.

10.1.1 No precision statement is made in regard to the other properties, because of the subjective nature of the observations.

10.2 *Bias*—The procedure in this test method for measuring changes in gloss, hiding, speckiness, agglomeration, coagulation, or color change has no bias because these values can be defined only in terms of the test method.

11. Keywords

11.1 freeze-thaw resistance; resistance to low-temperature cycling

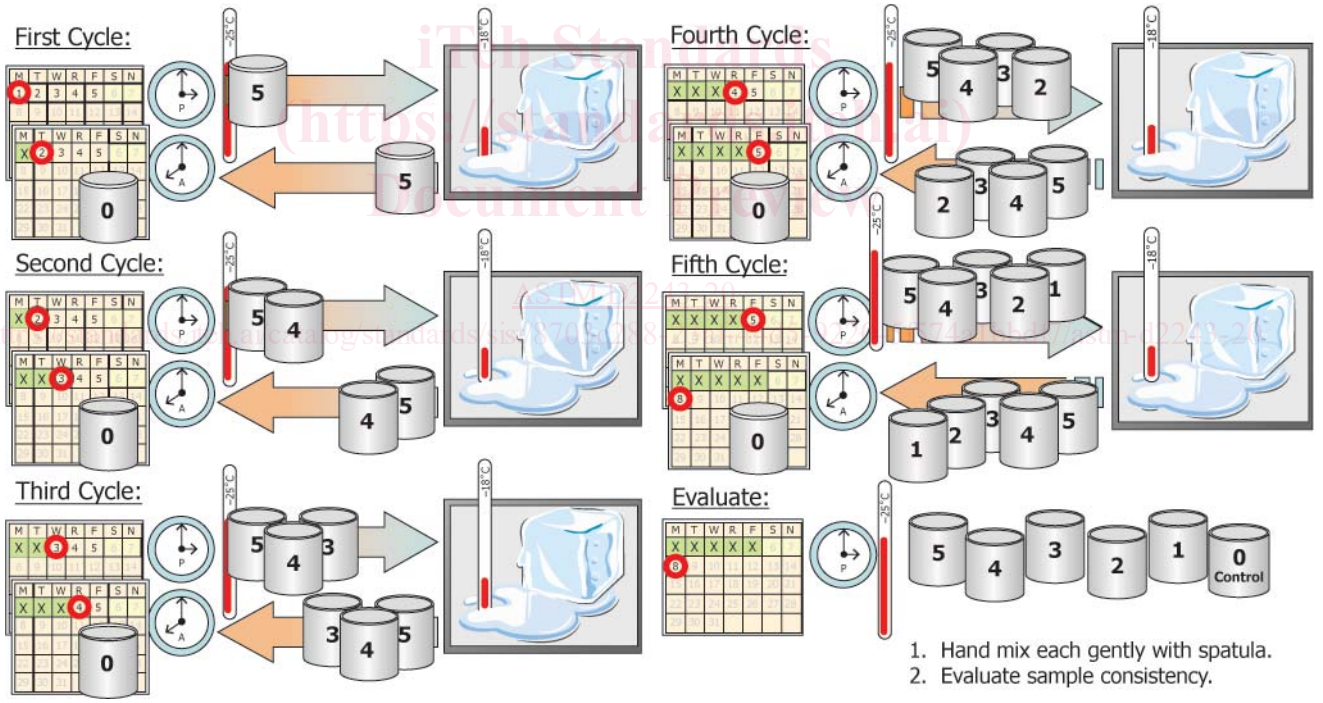
APPENDIX

(Nonmandatory Information)

X1. EXAMPLES OF STANDARD AND EXTENDED FREEZE/THAW CYCLES

X1.1 Fig. X1.1 is an example of standard 24-h cycle freeze/thaw cyclic testing (one way to do it).

X1.2 Fig. X1.2 is an example of using extended freeze/thaw workflow with an additional 24 h of thaw (total of 48 h per cycle).



Consistency Rating:
10: No chunks, can take KU reading.
8: No chunks, KU viscosity out of range.
6: No chunks, like dough or putty.
4: Small (<0.5 cm) chunks that won't redisperse; small curd cottage cheese.
2: Large (>0.5 cm) chunks that won't redisperse; large curd cottage cheese.
0: Hard, unable to stir. Like a hockey puck.

FIG. X1.1 Example of Standard 24-h Cycle Freeze/Thaw Cyclic Testing