



## Standard Test Method for Freeze-Thaw Resistance of Water-Borne Coatings<sup>1</sup>

This standard is issued under the fixed designation D 2243; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 562 Test Method for Consistency of Paints Measuring the Krebs Units (KU) Viscosity Using a Stormer-Type Viscometer<sup>2</sup>

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials By Rotational (Brookfield type) Viscometer<sup>2</sup>

### 3. Summary of Test Method

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

### 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 1 in. (25 mm) of air space between the sides of adjacent cans and capable of being maintained continuously at a temperature of  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ).

NOTE 1—Although a variation of the test chamber temperature of  $\pm 2^{\circ}\text{C}$  ( $\pm 3.5^{\circ}\text{F}$ ) is allowed, the test chamber temperature should be maintained as near  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) 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 D 562 or a Brookfield viscometer as described in Test Methods D 2196.

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.

### 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. Two such specimens are required for each test.

### 7. Exposure to Test Conditions

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

7.2 Place the second can, the paint under test, identified as the “test specimen”, in the chamber maintained at  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) in such a manner that it does not touch the walls or bottom of the chamber and so that free circulation of air around it 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 specimen in the chamber for 17 h and then remove and allow to stand for 7 h undisturbed at room

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.01.



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.).

## 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	4 = moderate
8 = very slight	2 = considerable
6 = slight	0 = complete failure

8.2 Stir the test and control paint by hand and measure their viscosities in accordance with Test Methods D 562 or D 2196. 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 D 562, specimens should be maintained at  $23 \pm 2^{\circ}\text{C}$  ( $73.5 \pm 3.5^{\circ}\text{F}$ ) until two successive readings agree within 5 g. 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 hiding, gloss, speckiness, agglomeration, coagulation, or color change. Rate changes as in 8.1.

## 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, in accordance with 8.2, and

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

## 10. Precision and Bias

10.1 *Precision*—The precision of this test method in regard to viscosities is as specified in Test Methods D 562 or D 2196, 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

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