



# Standard Test Method for Estimating Package Stability of Coatings for Ultraviolet Curing<sup>1</sup>

This standard is issued under the fixed designation D 4144; 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.

## 1. Scope

1.1 This method covers procedures for testing the package stability of coatings intended to be cured by ultraviolet radiation. One procedure is given for clear coatings and another for opaque fillers.

1.2 The values stated in inch-pound 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. Summary of Method

2.1 Specimens are placed in several containers, some of which are subjected to an elevated temperature while others are stored at room temperature. At specified intervals a specimen is checked for evidence of gelling. Clear materials are held in glass containers so they can be examined visually without opening to prevent contact with air which might inhibit polymerization. Opaque materials are checked by opening one can, probing the contents with a spatula to determine the extent of any polymerization, and then discarding that specimen.

## 3. Significance and Use

3.1 Coatings intended to be cured by ultraviolet radiation, especially those involving free radical chemistry, tend to polymerize during storage. It is of interest to determine how well a formulation resists this effect. Many factors influence the storage stability of a composition. The procedures described here are intended to improve the precision of determining this property. Because the effects of resins, monomers, photoinitiators, synergists, stabilizers, or pigments can alter the relation between elevated and room temperature stabilities, any correlation of performance at two different temperatures is possible only with a given formulation and, therefore, is useful only for quality control.

## 4. Apparatus

- 4.1 *Oven*, maintained at  $50 \pm 2^\circ\text{C}$ .
- 4.2 *Glass Jars*, wide-mouth, 4-oz (115-mL), with 38-mm closures.
- 4.3 *Cans*, lined, 4-oz (115-mL), friction top, with lids.
- 4.4 *Spheres*, glass or porcelain, 7 to 10-mm diameter.

## 5. Procedure

### 5.1 Clear Coatings:

5.1.1 Fill three 4-oz (115-mL) wide-mouth jars to  $\frac{1}{4}$  in. (6 mm) from the top. Add a small glass or porcelain sphere to each container and put the lids on tightly.

5.1.1.1 The amount of head space in a jar or can is critical because the volume of air in contact with the sample has an effect on the rate of polymerization. The stability is also related to the ratio of the area of liquid-air interface to the volume of liquid.

5.1.2 Put two jars in an oven at  $50 \pm 2^\circ\text{C}$ . Retain the third at a temperature,  $25 \pm 2^\circ\text{C}$ , and in the dark.

5.1.3 Check an oven jar daily but do not open or invert. Rather, tip slightly, no more than  $30^\circ$ , to determine the extent of polymerization by noting the mobility of the sphere. When gelling is noticed, check the second jar to confirm.

5.1.4 Record the duration of the test in days. Indicate the last day the sphere is mobile followed by the first day it is immobile, and if the days are not consecutive, why the interval occurred.

5.1.5 Check the jar stored at room temperature every week but do not open or invert. Tip slightly, no more than  $30^\circ$ , to determine if the sphere is immobile.

5.1.6 Record the number of weeks not gelled followed by the first week the sphere is immobile.

### 5.2 Pigmented (Opaque) Coatings:

5.2.1 Fill twelve 4-oz (115-mL) lined cans to  $\frac{1}{4}$  in. (6 mm) from the top and put the lids on tightly.

5.2.1.1 See 5.1.1.1.

5.2.2 Put six of the cans in an oven at  $50 \pm 2^\circ\text{C}$ . Retain six cans at ambient temperature,  $25 \pm 2^\circ\text{C}$ .

5.2.3 After one day remove one can from the oven, open, and probe to the bottom to determine if gelling is beginning. Discard the can after the test. Check one of the remaining cans on the 2nd, 4th, 8th, 16th, and 32nd days and discard after testing. By starting on a Monday all the testing will fall on

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normal working days.

5.2.4 Record the condition of the specimen each day tested, indicating the fractional amount of any gelled material present.

5.2.5 After one week check one of the cans held at ambient temperature by opening and probing to the bottom to determine if gelling is beginning. Discard the can after the test. Check one of the remaining cans after 2, 4, 8, 16, and 32 weeks.

5.2.6 Record the condition of the specimen after each test and indicate the fractional amount of any gelled material present.

## 6. Report

6.1 Report whether the material was clear or pigmented and the length of time it was stable as indicated by the occurrence of gelation at both ambient and elevated temperatures. Report the age of the material when the test began, if it is known.

## 7. Precision and Bias

### 7.1 Precision:

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### 7.1.1 Clear Coatings:

7.1.1.1 At the elevated temperature, four of five cooperators reported a clear coating without inhibitor gelled on the 8th or 9th day. Four of five cooperators reported a coating with inhibitor gelled on the 32nd day; the fifth reported gelation on the 18th day.

7.1.1.2 At room temperature four of four cooperators reported no gelling at 32 weeks, with or without inhibitor.

### 7.1.2 Pigmented Coatings:

7.1.2.1 At the elevated temperature six of six cooperators reported a filler gelled at 8 days.

7.1.2.2 At room temperature four of four cooperators reported various degrees of gelation after 32 weeks.

7.2 *Bias*—Since there is no accepted reference material for this test method, bias cannot be determined.

## 8. Keywords

8.1 stability; ultraviolet cure