



Designation: D 4144 – 94 (Reapproved 1999)

Standard Test Method for Estimating Package Stability of Coatings for Ultraviolet Curing¹

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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° , 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° , 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.

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 D01.24 on Physical Properties of Liquid Paints and Paint Materials.

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