



Standard Test Method for Heat Stability of Hot-Melt Adhesives¹

This standard is issued under the fixed designation D 4499; 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.

1. Scope

1.1 This test method covers the evaluation of hot-melt adhesives with respect to the change in properties that occurs while the adhesive is aged in the molten state during hot-melt application. Melt properties monitored are viscosity, color, skin formation, and phase separation.

1.2 The values stated in SI units are to be regarded as the standard.

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 907 Terminology of Adhesives²

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)^{3, 4}

D 3236 Test Method for Apparent Viscosity of Hot-Melt Adhesives and Coating Materials⁵

3. Terminology

3.1 *Definitions*—Several terms in this test method are defined in accordance with Terminology D 907.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *color*—the aspect of the appearance of an object dependent upon the spectral composition of the incident light, the spectral reflectance or transmittance of the object, and the spectral response of an observer.

3.2.2 *melt viscosity*—measure of the ratio of shear stress to shear rate when the hot-melt is in its molten state.

3.2.3 *phase separation*—formation of a second liquid portion from a previously homogenous liquid over time.

3.2.4 *skin formation*—appearance of a relatively loose layer at the surface of a polymeric material.

4. Summary of Test Method

4.1 Samples of hot-melt adhesives are conditioned at the adhesive manufacturer's recommended application temperature for a conditioning cycle that corresponds to the expected residence time during which the adhesive will be held molten in the storage and application equipment. If so specified by the adhesive manufacturer, the molten adhesive will be protected against exposure to air. The total residence time is divided into two or three intervals at which points the aged adhesive color and melt viscosity are measured and compared to the initial adhesive color and viscosity. Prior to measuring the melt viscosity and color, the adhesive is subjectively evaluated for evidence of phase separation and surface skinning. Separate adhesive samples are conditioned for each test period.

4.2 The test report includes percent change in melt viscosity, percent of adhesive surface skinned, percent change in Gardner color, and description of phase separation, if any.

5. Significance and Use

5.1 Hot-melt adhesives must generally be applied at relatively high temperatures in order that the adhesive viscosity is low enough that it can be readily handled in typical hot-melt equipment. Changes in the adhesive caused by exposure to the severe environment required during hot-melt processing will disrupt the adhesive application process and may affect the quality of the adhesive bond. Changes in adhesive viscosity may affect the quantity of adhesive dispensed by the applicator. Phase separation or changes in viscosity and color may be an indication of changes occurring in the adhesive which could affect the quality of the adhesive joint. A skin formed on the adhesive may eventually clog the applicator nozzle or die.

5.2 The data generated in this test procedure are comparative in nature. Results can be useful in observing batch-to-batch variation or relative thermal stability of alternate adhesive formulations. Results can also be useful in selecting application equipment and establishing operating conditions for satisfactory handling of a particular hot-melt adhesive.

5.3 Round-robin testing showed that the reproducibility of this test method can depend strongly on the characteristics of the adhesive being tested. Results showed that, for the particular adhesives tested in the round robin, the test method was well suited for testing the heat stability of the adhesives based on an EVA copolymer, a polyolefin, and a polyamide. However, because of poor reproducibility, the test method proved to be unsuitable for assessing the heat stability of the adhesive

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² *Annual Book of ASTM Standards*, Vol 15.06.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ Visual color standards such as the Pantone Color System can be used in place of the Gardner Color System with pigmented adhesives.

⁵ *Annual Book of ASTM Standards*, Vol 05.02.