



Standard Test Method for Determining the Open Time of Hot Melt Adhesives (Manual Method)¹

This standard is issued under the fixed designation D 4497; 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 manual determination of the open-time range of hot-melt adhesives under the stated conditions.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses are provided 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 907 Terminology of Adhesives²

E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

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 *open time*—the time available between application of the adhesive to the first substrate and the mating of the second substrate which still results in a minimum of 50 % fiber tear when tested at ambient conditions.

4. Summary of Test Method

4.1 In this test method, open time is determined by bonding together with the adhesive, pieces of standard paper at intervals of 5 s sequentially after the molten adhesive has been applied using a heated film applicator on one surface. The bonded

specimens are examined later to determine the longest time period, between application of the adhesive to one surface and mating with a second surface, which allows substantial bonding.

5. Significance and Use

5.1 This test method allows comparing the open time of several hot melt adhesives.

5.2 This test method is not intended to provide the open-time observed for a specific adhesive in a specific manufacturing operation. This test method can be used to compare the performance of adhesives which are represented to be identical or to rank the open-time of a group of hot melt adhesives.

6. Apparatus

6.1 *Oven*, which meets the precision required by the procedure, should be used to heat the adhesive sample and the applicator.

6.2 *Film Applicator*— Multiple clearance 0.125 to 1.25 mm (5 to 50 mil) square P.G. and T. Number 2 Film Applicator⁵ capable of providing an applied film of adhesive having a nominal width of 50 mm (2 in.).

6.3 *Hand Thermometer*, preferably metal, capable of measuring the temperature of the hot-melt with an accuracy of $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$).

6.4 *Primary Standard Substrate*, 200 by 280 mm (8 by 11 in.), NBS Standard Reference Material 1810 (Liner Board).⁶ Substrates are to be conditioned as described in Specification E 171.

6.5 *Secondary Standard Substrate*, 125 mm (5 in.) length in the material direction cut to 19 mm (0.75 in.) wide, NBS Standard Reference Material 1810 (Liner Board).⁵

6.6 *Glass Beakers* (600 mL), for melting the adhesive.

6.7 *Watchglass*, to cover beaker.

6.8 *Beakers, Tongs, and Protective gloves*, for handling hot vessels and applicators.

6.9 *Transparent Pressure Sensitive Tape*.

6.10 *Stop Watch or Other Timer*, graduated to at least 0.5 s.

¹ This test method is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.50 on Hot Melt and Pressure Sensitive Adhesives.

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

³ *Annual Book of ASTM Standards*, Vol 15.09.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Film Applicator No. 2 is available from Precision Gage and Tool Company, 28 Volkenand Street, Dayton, OH 45410.

⁶ Available from National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

6.11 *Weighted Rectangular Block*, having at least one smooth and flat wood face, 50 by 12.5 mm (2 by 0.5 in.) weighing 100 g (3.5 oz), the mass being uniformly distributed.

6.12 *Micrometer or Thickness Gage*, capable of measuring to the nearest 0.025 mm (1 mil).

7. Sampling

7.1 The test sample should be representative of the adhesive being tested.

8. Procedure

8.1 Add 300 ± 10 g of the adhesive to the 600-mL glass beaker and cover with a watch glass. Place the beaker containing the adhesive into the oven and heat until at the application temperature recommended by the manufacturer.

8.2 Concurrently heat the film applicator in the same oven to condition it to the sample temperature as in 8.1.

8.3 Perform testing in a relatively draft-free room. While the sample is melting, prepare the substrates for testing as shown in Figs. 1 and 2.

8.3.1 Tape the long sides of a piece of primary standard substrate (see 6.4), minimum sheet size 100 by 280 mm (4 by 11 in.), to a flat smooth surface of low thermal conductivity, specifically to a larger piece of chipboard (such as from the back of a tablet). Then tape the chipboard to an underlying surface.

8.3.2 Place the strips of the secondary standard substrate (see 6.5) across the width of the primary substrate, as shown in Fig. 1, leaving approximately 6 mm ($\frac{1}{4}$ in.) between the strips. The top strip should be approximately 75 mm (3 in.) below the top edge of the primary substrate. Using pressure sensitive tape, attach one end of the strips to the primary substrate. Fold the unattached end of each strip back over the attached end so that the primary substrate is exposed for application of the adhesive (see Fig. 2).

8.4 Remove the sample from the oven, and by stirring with the metal thermometer, quickly verify that the temperature is within $\pm 3^\circ\text{C}$ ($\pm 5^\circ\text{F}$) of the manufacturers recommended application temperature.

8.5 Quickly remove the adhesive applicator from the oven, and center it above the top strip of the secondary substrate. The 0.5 mm (20-mil) gap should face down and be near the top end of the primary substrate.

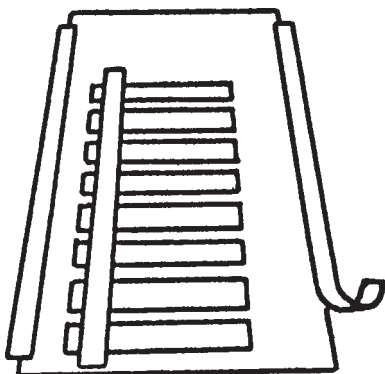


FIG. 1 Secondary Substrate Strips on Primary Substrate Before Hot Melt Application

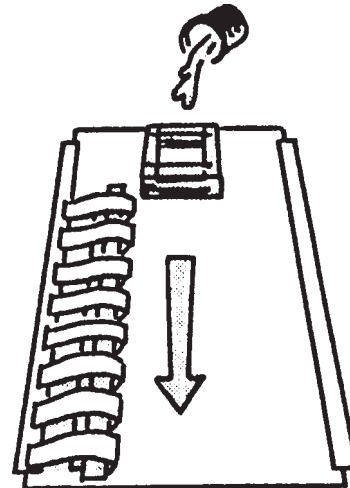


FIG. 2 Strips Drawn Back to Allow Hot Melt Application

8.6 Fill the applicator reservoir to approximately a $\frac{1}{4}$ -in. level with conditioned hot adhesive.

8.7 Move the applicator and the contained molten adhesive with one smooth motion over the length of the paper taking approximately 2 s to complete the drawdown.

NOTE 1—Approximately 2 s is satisfactory. The purpose is to standardize on a reasonable rate and eliminate an excessively fast or slow drawdown which will affect the results.

NOTE 2—Trial applications should be made to determine the proper applicator gap to lay down a specified film thickness. Normally, a gap of 0.50 mm (20 mil) will lay down a film of 0.25 mm (10 mil). Variations in applied film thickness will occur because of differences in the hot flow properties of the adhesive.

8.8 Immediately after the film has been drawn down, start the timer.

8.9 At 5 s on the timer, or a longer time if it is known that an adhesive will have an open time longer than about 30 s, lay the top piece of secondary substrate across the cooling film, and continue sequentially at 5-s intervals. Use the weighted block to apply immediately a momentary load of 5-s duration to press the strips onto the adhesive, moving the block to successive strips as they are laid onto the adhesive. See Fig. 3. The wood face of the block faces the substrate. Repeat this operation until it is obvious the adhesive film has solidified and will not bond to the secondary substrate.

8.10 Allow the adhesive film to cool at ambient temperature for 30 to 60 min before measuring.

8.11 Measure the combined thickness of the adhesive film and the two substrates. Subtract the thickness of the two substrates to determine the nominal adhesive thickness to the nearest 0.025 mm (1 mil).

8.12 With a uniform motion, peel off each strip of secondary substrate at an angle of 90° to the surface. Take about 10 s to peel off each strip. If the paper tears near the initial edge, repeat the peel from the opposite end of the strip. Record the percent area of fiber tear for each strip and the time in seconds at which that strip was laid onto the cooling adhesive.

8.13 Repeat the entire test procedure in 8.1-8.12 for a total of three determinations.

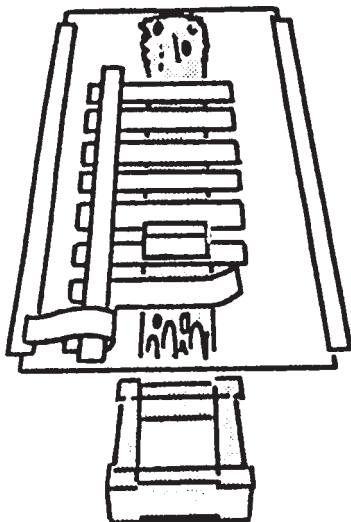


FIG. 3 Strips Successively Placed onto Hot Melt

9. Report

9.1 Report the mean open-time of the three replicate determinations as the longest time, to the nearest 5 s, at which a minimum of 50 % fiber tear is obtained.

- 9.2 Report the ambient temperature and relative humidity.
- 9.3 Report nominal adhesive thickness, to the nearest 0.025 mm (1 mil), as the mean of the three replicate determinations.
- 9.4 Report the application temperature used (see 8.4).

10. Precision and Bias

10.1 The precision and bias of this test method is based upon the testing of five typical hot melt adhesives by six laboratories. Statistical analysis of these data is tabulated in Appendix X1.

10.2 The within-laboratory, single operator standard deviation of this test method for determining the open-time of a hot melt adhesive is equal to or less than 2.6 s. This was determined for typical hot melt adhesives having mean open-times by this test method of 20.8 s or less. The between-laboratory standard deviation including within- and between-laboratory variability is equal to or less than 8.4 s.

11. Keywords

- 11.1 application temperature; open time

APPENDIX

(Nonmandatory Information)

X1. STATISTICAL ANALYSIS FOR TEST METHOD D 4497

TABLE X1.1 Hot Melt Adhesive Open Time—Basic Data^A

Laboratory	Adhesive Type									Polypropylene			Polyamide		
	Ethylene-Vinyl			Polyester			Polyethylene								
	Acetate														
	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)
1. Open Time, s	5	10	5	5	5	5	20	20	15	30	30	35	10	10	15
Temperature, °F	350	350	350	425	425	425	350	350	350	350	350	350	425	425	425
Film Thickness, mil	10	10	10	12	12	12	10	10	10	12	11	9	11	11	11
2. Open Time, s	5	5	5	40 ^B	40 ^B	40 ^B	15	15	15	20	20	20	20	20	20
Temperature, °F	349	348	352	420	422	425	352	351	352	321	326	324	420	421	420
Film Thickness, mil	11	11.5	11	11	12	12.5	10	10.5	10	12.5	11	11	10	10	10
3. Open Time, s	10	10	10	5	5	5	20	20	20	30	30	30	20	20	25
Temperature, °F	350	350	350	425	425	425	350	350	350	325	325	325	425	425	425
Film Thickness, mil	8	9	9	9	9	9	9	9	8	9	9	9	9	9	9
4. Open time, s	10	10	10	10	15	10	15	15	15	20	15	15	15	20	20
Temperature, °F	350	350	350	425	425	425	350	350	350	325	325	325	425	425	425
Film Thickness, mil	10	10	10	11–12	12	11–12	9–10	10	10–11	10–11	10–11	10–11	10–11	10–11	10–11
5. Open Time, s	10	10	10	5	10	5	15	15	10	10	10	15	20	25	20
Temperature, °F	350	350	350	425	425	425	350	350	350	350	350	350	425	425	425
Film Thickness, mil	6	5	7	8	8	9	4	3	5	3	5	5	8	8	7
6. Open Time, s	5	5	5	5	5	5	15	15	15	15	15	15	10	15	10
Temperature, °F	350	350	350	425	425	425	350	350	350	350	350	350	425	425	425
Film Thickness, mil	9	9	8	12	12	13	8	9	9	10	9	10	9	9	10

^A Values reported as <5 s are entered into the statistical analysis as 0.

^B Outlier values were eliminated from statistical analysis according to Practice E 691.

TABLE X1.2 95 % Repeatability and Reproducibility Intervals

Adhesive Type	95 % Repeatability Intervals Within Laboratory, s	95 % Reproducibility Intervals Between Laboratories, s
Ethylene-Vinyl Acetate	3.3	7.6
Polyester	7.5	12.7
Polyethylene	4.7	8.2
Polypropylene	5.8	23.8
Polyamide	7.5	14.7

TABLE X1.3 95 % Confidence Intervals

Adhesive Type	s
Ethylene-Vinyl Acetate	±3.1
Polyester	±6.8
Polyethylene	±4.4
Polypropylene	±5.3
Polyamide	±6.9

TABLE X1.4 Precision Estimates of Open Time of Hot Melt Adhesives

Adhesive Type	Mean Open Time, s					
	S_r	S_L	V_r %	V_L %	S_R	
Ethylene-Vinyl Acetate	7.8	1.2	2.4	15	31	2.7
Polyester	4.7	2.6	3.7	56	78	4.5
Polyethylene	16.1	1.7	2.3	10	14	2.9
Polypropylene	20.8	2.0	8.1	10	39	8.4
Polyamide	17.5	2.6	4.4	15	25	5.2
Average	21	38	...

where:

- S_r = estimated repeatability standard deviation within-laboratories for that adhesive,
- S_L = estimated between-laboratory standard deviation for that adhesive,
- V_r = estimated standard deviation within-laboratories as a percent of the mean open time,
- V_L = estimated between-laboratories standard deviation as a percent of the mean open time, and
- S_R = between-laboratory estimate of standard deviation including within- and between-laboratory variability.

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