Designation: D 6463/D 6463M – 06

Standard Test Method for Time to Failure of Pressure Sensitive Articles Under Sustained Shear Loading[1]

This standard is issued under the fixed designation D 6463/D 6463M; 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 ability of a pressure sensitive article (for example, tape, label, sticker, etc.) to remain adhered to a stainless steel panel under a constant load applied parallel to the bonding surface of the pressure sensitive article.

1.2 Values stated in either SI or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents, therefore, each system must be used independently without combining values in any way.

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:* [2]

A 666 Specification for Annealed or Cold-Worked Austenitic Stainless Steel Sheet, Strip, Plate, and Flat Bar

D 907 Terminology of Adhesives

D 3654/D 3654M Test Methods for Shear Adhesion of Pressure-Sensitive Tapes

D 5750/D 5750M Guide for Width and Length of Pressure-Sensitive Tape

E 171 Specification for Atmospheres for Conditioning and Testing Flexible Barrier Materials

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

3. Terminology

3.1 Many of the terms found in this test method are defined in Terminology D 907.

4. Summary of Test Method

4.1 This test method consists of bonding a standard area of a pressure sensitive article to a stainless steel panel. The bonded construction is allowed to dwell in a constant temperature and humidity environment for 30 min. The construction is then mounted vertically. A standard mass of 500 g [1.1 lb] is attached to the pressure sensitive article and allowed to act until the construction separates or until 3000 min elapse. If there is no separation after 3000 min, the test is repeated on a fresh specimen with a mass of 1000 g [2.2 lb] and allowed to act until separation occurs or until 3000 min elapse. If no separation occurs, perform Procedure C of Test Method D 3654/D 3654M for shear holding power to stainless steel at 49°C [120°F]. The test result is the time in minutes to separation.

5. Significance and Use

5.1 This test method measures the cohesive strength of the adhesive on a pressure sensitive article. The time to failure can predict end use properties of pressure sensitive articles such as

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.50 on Hot Melts, Pressure Sensitives, and Archival Adhesives.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

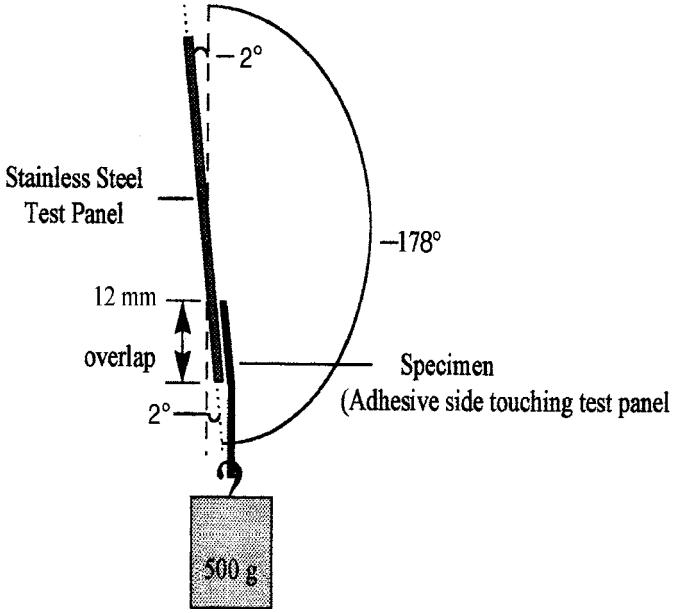

FIG. 1 Test Panel with Specimen and Mass (Side View)

edge ooze from cold flow; trimming, slitting and die cutting quality; telescoping in tapes; ability to seal packages; and vertical holding power.

5.2 This test method is suitable for quality control, development and applications testing.

5.3 If the adhesive is not already coated, the adhesive can be coated on smooth, clear polyester (PET) film backing 0.05 mm [0.002 in.] thick. The recommended dry adhesive thickness is 0.025 mm [0.001 in.].

5.4 Humidity has a strong effect on time to failure for many pressure sensitive articles. Therefore, humidity should be controlled in accordance with 8.1.2.

6. Apparatus

6.1 *Specimen Cutter* [3,4]— The specimen cutter shall hold two single-edge razor blades in parallel planes, a precise distance apart, to form a cutter of exact specimen width. A cutter of 12 mm [0.5 in.] cutting width, shall be available or appropriate alternates, that will not cause edge damage.

6.1.1 The 12 mm [0.5 in.] cutter shall consist of a 12 mm [0.5 in.] thick by 200 mm [8 in.] length of aluminum bar stock 12 mm [0.5 in.] wide. The edges for about 125 mm [5 in.] from one end shall be rounded slightly to form a handle. The width of the bar for 75 mm [3 in.] shall be narrowed to exactly 12 mm [0.5 in] minus the thickness of a single-edge razor (one of two used as cutting edges). The razor shall be held in position using side plates. The end of the cutter shall be cut away at a 45° angle to expose the cutting edges at one end of the blades. The edges shall be separated by 12 ± 0.10 mm [0.5 ± 0.005 in.].

6.2 *Dispensing System*, for solvents, such as a wash bottle.

6.3 *Test Panel* [4]—No. 302 or 304 stainless steel sheet, not less than 1.1 mm [0.43 in.] thick, having a bright annealed finish, in accordance with Specification A 666. The panel should be 25 by 25 mm [1.0 by 1.0 in.] or larger, as best suited to the test stand (Fig. 1). The surface roughness height shall be 50 ± 25 nm [2.0 ± 1.0 µin.] arithmetical average deviation from a mean line. One or both of the panel ends shall be ground to form a 90° angle with the panel surface. Panels showing stains, discolorations, or numerous scratches are not acceptable. New panels should be cleaned before use as described in 10.1, except with ten washes of the final solvent. Between uses, the panels test surface shall be protected from scratches and contamination, and the panels stored at conditions described in Section 8.

6.4 *Roller*, mechanically or hand-operated.[4]

6.4.1 A steel roller 85 ± 2.5 mm [3.25 ± 0.1 in.] in diameter and 45 ± 1.5 mm [1.75 by 0.05 in.] in width, covered with rubber approximately 6 mm [0.25 in.] in thickness, having a Shore scale A durometer hardness of 80 ± 5. The surface shall be a true cylinder void of any convex or concave deviations. The mass of the roller shall be 2040 ± 45 g [4.5 ± 0.1 lb].

6.4.2 No part of the apparatus shall increase the mass of the roller during use. The roller shall move either mechanically or by hand at the rate of 10 ± 0.2 mm/s [24 ± 0.5 in./min.].

6.5 *Test Stands and Ancillary Apparatus*:

6.5.1 *Test Stand* [4]—A test stand that will hold the test panel with the specimen applied at an angle of 2.0 ± 0.5° from vertical, so that when the mass is acting on the test specimen, no peel forces will be exerted on the specimen.

6.5.2 *Clamp/Clip/Cardboard Chip*—that will allow attachment of the mass to the specimen, distributing the load equally across the specimen width.

[3] These widths correspond to the primary metric (SI) units described in Guide D 5750/D 5750M. These so-called " modular metric" units generally are used throughout the world. If it is desirable to test slightly different widths, this should be noted per 12.1.8 and calculations per 11.1 must account for the difference.

[4] The sole source of supply of the apparatus known to the committee at this time is Chemsultants International, 9349 Hamilton Drive, Mentor, OH 44061–1118. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] may attend.

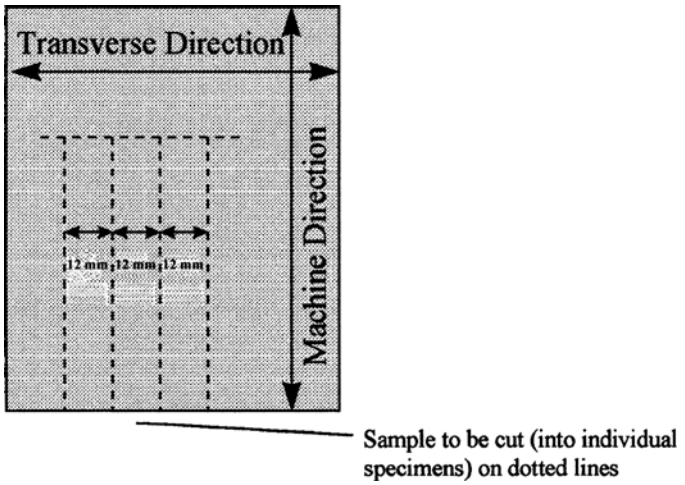

FIG. 2 Typical Sample of a Pressure Sensitive Article (Schematic for Sample Preparation)

6.5.3 *Mass*—a 500 ± 5 g [1.1 ± 0.01 lb] and a 1 000 ± 5 g [2.2 ± 0.01 lb] with a hook on top.

6.5.4 *Timing System*—to measure the interval in minutes, between the application of the load to the specimen and its separation form the panel.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals should be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening accuracy of the determination.

7.2 *Solvents*:

7.2.1 Any of the following solvents may be used for cleaning:

7.2.1.1 Diactone alcohol non-residual, technical grade or better,

7.2.1.2 Methanol (95 %),

7.2.1.3 Methyl ethyl ketone (MEK),

7.2.1.4 n-Heptane, or

7.2.1.5 Acetone.

7.2.2 Before selecting or using these solvents for cleaning test panels, be sure to read and follow all precautions on the chemical Material Safety Data Sheets (MSDS) and consult with Environmental, Health, and Safety (EHS) professionals.

7.3 *Cleaning Material*—Absorbent, surgical gauze, cotton wool or tissue may be used. To be suitable, materials must be lint-free during use, absorbent, contain no additives that are soluble in the solvents listed in 7.2 and made exclusively form virgin materials.

8. Conditioning

8.1 *Testing Room*—Condition and test samples in a controlled temperature and humidity room is described below:

8.1.1 Control the temperature to 23.0 ± 2.0°C [73.4 ± 3.6°F] as specified in Specification E 171.

8.1.2 Control the relative humidity to 50 % RH± 2 % RH. The humidity range specified in Specification E 171, 50 % ± 5 % RH, is too large for this test.

8.1.2.1 Two independent studies were conducted on the effect of relative humidity on time to failure of pressure sensitive articles under sustained shear loading. For four acrylate adhesive tapes, the time to failure decreased by 5.8, 5.5, 8.4 and 6.1 % for each 1 % increase in relative humidity. For each sample, the rate of humidity decrease was determined at 50 % RH. This high sensitivity to small humidity changes requires that humidity be controlled during the test.

8.2 Condition samples in a controlled temperature and humidity room, as described in 8.1, for at least 24 h before cutting the samples into test specimens (Sample Preparation, See Section 9).

9. Sample Preparation

9.1 Cut sample into 12 mm [0.5 in.] wide strips with the long dimension in the machine direction of the pressure sensitive article. The dimensions of the shear stand will dictate the specimen length. The mass drop distance (the distance between the bottom of the mass and the timer stop plate) must be a minimum of 12 mm [0.5 in.] and should be such that the shear stand does not transmit shock forces to the specimens that remain hanging when the mass drops. Cut at least three specimens for each pressure sensitive article to be tested (Fig. 2).

NOTE 1—If the sample to be tested incorporates PVC (or any other type of facestock with a tendency to elongate under load) as a facestock, the sample may be reinforced by overlaminating it with a non-elastic film, such as PET, prior to sample cutting. This minimizes the influence of the facestock on the time to failure.

10. Procedure

10.1 Dispense one of the solvents listed in 7.2.1 onto the panel, wiping to dryness with fresh absorbent cleaning material (see 7.3). Repeat for a total of three washes. The final cleaning shall be with an alternate solvent, either MEK or acetone. Do not touch cleaned panel surfaces with fingers. The cleaned panel should be allowed to dry at standard conditions for at least 10 min. If cleaned panel is not used within 10 h, it should be recleaned.

NOTE 2—All operations with solvents should be conducted in a well-ventilated hood.

NOTE 3—Discard panels showing stains, discoloration, or many

TABLE 1 Shear Test Precision Data (All Data in Minutes)

Material	Average, min.	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
A	278.7	34.9	72.4	97.8	202.8
B	742.6	48.8	163.5	136.7	457.7

scratches. During storage, panels should be protected from damage or contamination.

10.2 Attach the adhesive surface of one end of the specimen to a clip or piece of cardboard to distribute the force of the mass evenly across the width of the specimen. Reinforce that end of the specimen with a piece of tape, a staple or both so that it will hold the mass securely.

10.3 Center the specimen on the bottom end of the clean test panel. Remove any release liner and apply the specimen to the panel without added pressure. The specimen covers 12 by 12 mm [0.5 by 0.5 in.] on the test panel.

10.4 Adhere the specimen to the panel by rolling the rubber covered roller over the covered area four times, twice back and forth, at a velocity of 600 mm [24 in.]/min. Either a hand or a mechanical roller may be used.

10.5 Insert the panel and specimen into the test stand holder and wait 30 min before applying the standard mass.

10.6 Gently apply the 500 g [1.1 lb] mass to the specimen so that no impact force or oscillations are applied. The drop distance should be as described in 9.1.

10.7 Record the time elapsed in minutes when the specimen separates from the test panel.

10.7.1 If no separation occurs within 3000 min with the 500 g [1.1 lb] mass, repeat the test with a fresh specimen and attach the 1000 g [2.2 lb] mass in 10.6.

10.7.2 If no separation occurs within 3000 min with the 1000 g [2.2 lb] mass, terminate the test and use the conditions specified in Procedure C of Test Method D 3654/D 3654M.

10.8 Repeat the procedure at least two additional times for each sample to be tested for a total of three replicates.

11. Calculation

11.1 Calculate the average time to failure (of at least three specimens) in minutes and the standard deviation for each sample.

12. Report

12.1 Report the following information:

12.1.1 Adhesive identification and type, if known,

12.1.2 Cleaning solvent(s) used for cleaning test panel,

12.1.3 Adhesive coat weight or thickness, if known,

12.1.4 Facestock material and release liner used, if known,

12.1.5 Temperature and relative humidity of testing room,

12.1.6 Average time to failure (of at least three specimens) and standard deviation,

12.1.7 Failure mode - adhesion, cohesion or transfer. Cohesion is the preferred failure mode for this test, and

12.1.8 Any deviation from the procedure as written.

13. Precision and Bias

13.1 An interlaboratory study of this test method using the original ASTM roll-down procedure, two passes of the roller at 300 mm [12 in.]/min., was conducted in accordance with Practice E 691. Seven laboratories reported three test results on each of two adhesive tapes. Results of the repeatability study are summarized in Table 1. Repeatability and reproducibility of this test method with the new harmonized standard roll-down will be conducted and available on or before December 12, 2009. All other information herein refers to the original study.

13.2 The precision information given below was calculated for the comparison of the average of three individual test determinations as specified in the test method.

13.3 The terms repeatability limit and reproducibility limit are used as specified in Practice E 177.

13.4 *Repeatability*—The repeatability of this test method was an increasing function of the value of the test result. See Table 1.

13.5 *Reproducibility*—The reproducibility of this test method was an increasing function of the value of the test result. See Table 1.

13.6 *Bias*—No measurement of bias is possible with this test method because an accepted reference or referee value is not available.

14. Keywords

14.1 adhesive; cohesion; pressure sensitive adhesive; pressure sensitive article; shear; sustained shear loading; time to failure