Designation: D 1916 – 93 (Reapproved 1997)

Standard Test Method for Penetration of Adhesives[1]

This standard is issued under the fixed designation D 1916; 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 determination of the penetration under pressure of adhesives used in systems where at least one of the adherends is porous.

1.2 *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 Adhesives[2]

3. Terminology

3.1 *Definitions*—Many terms in this test method are defined in Terminology D 907.

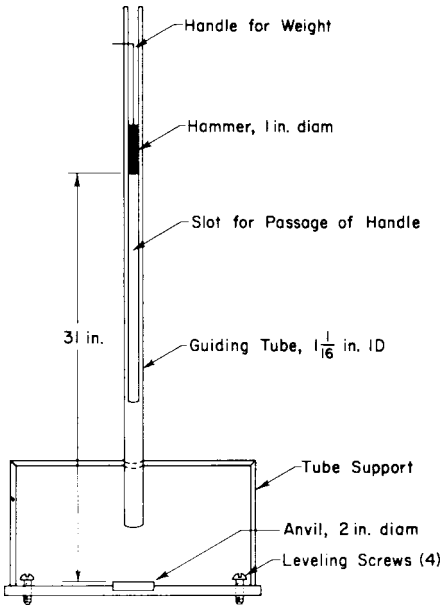
3.1.1 *adhesive penetration, n*—the entering of an adhesive into a porous adherend.

4. Significance and Use

4.1 This test method is useful in selecting adhesive systems for a particular application, and in modifying adhesive systems to obtain maximum performance without excessive penetration of the adhesive into the substrate of the adherends. It is particularly adaptable to use with starch or starch-base adhesives commonly used in the paper converting industry. Further development may well result in its usefulness with other types of adhesives.

5. Apparatus

5.1 *Penetration Tester*, as shown in Fig. 1, constructed so as to allow a section of round steel bar stock 25 mm (1 in.) in diameter and weighing 700 g (the hammer), to fall freely through a distance of 0.8 m (31 in.) upon a steel disk (the anvil) 6 mm (¼ in.) thick and 51 mm (2 in.) in diameter, in such a manner as to strike the anvil at right angles. Dimensions of the guiding tube and of the section of bar stock, as shown in Fig. 1, permit free fall of the bar stock without undue sideways movement. Cut the bottom end of the bar stock at right angles to the longitudinal axis, within 0.051-mm (0.002-in.) tolerance, and finish to 32 microfinish.



Metric Equivalents				
in.	1	1 1/16	2	31
mm	25	27	51	787 (0.8 m)

FIG. 1 Penetration Tester

5.2 *Filter Paper*, coarse, rapid-filtering type, cut into 51-mm (2-in.) disks.

5.3 *Syringe*, hypodermic, 1-mL capacity, without needle.

5.4 *Densitometer.*[3]

5.5 *Compensating Planimeter*, unit range 0.01 in.^{2} (6.5 mm^2).

6. Reagent

6.1 *Staining Reagent*, suitable for the adhesive being tested. For adhesives containing starch, an iodine solution containing 2.0 g of potassium iodide and 0.20 g of iodine in 3 L of distilled water is appropriate. Some adhesives that are basic in pH must be stained with phenolphthalein or other suitable indicator solution.

[1] This test method is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.10 on Working Properties.

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

[3] A MacBeth-Ansco Color Densitometer, Model 12, has been found satisfactory for this purpose.

7. Procedure

7.1 Place five disks of filter paper, stacked one on top of another, upon the anvil at the bottom of the tube. Apply 0.1 mL of the adhesive to be tested to the center of the bottom end of the hammer (bar stock) using the 1-mL syringe (Note 1). If the hammer has been inverted for this purpose, reinvert the hammer so that the adhesive is now on the bottom and place in the testing apparatus with the handle of the hammer resting in the notch provided for it (Note 2).

NOTE 1—The hammer may be inverted for this purpose.

NOTE 2—Inversion of the hammer without loss of adhesive may require some practice. If any adhesive is lost during this step, restart the test.

7.2 Thirty seconds after the adhesive is applied to the hammer, release the hammer from this position, allowing it to fall freely and strike the anvil.

NOTE 3—Application of the adhesive to the hammer and release of the hammer is accomplished quickly in order to reduce loss of solvent.

7.3 Immediately raise the hammer, remove and separate the stack of filter papers, then clean the hammer with a damp cloth or sponge.

7.4 Stain the filter papers by applying a slight excess of an appropriate staining solution. Allow the stained papers to air-dry for 1 h prior to evaluating density with the densitometer. A minimum of five different tests of each adhesive is recommended to obtain significant and checkable results.

NOTE 4—Variables making multiple readings necessary include differences in porosity and direction of fibers in filter paper. These factors cause some distortion of color intensity within a spot and spot contour from level to level.

7.5 Carefully outline the stained areas on each level with a very sharp No. 2 lead pencil. Do not indent the paper, and avoid touching the areas to be read with the densitometer.

7.6 Allow the densitometer to reach equilibrium and use a blue color filter having a peak response of 436 nm for iodine stain (Note 5). Take five densitometer readings, distributed more or less symmetrically, on the stained area of each of the five disks of filter paper and average the results obtained on each disk (Note 6).

NOTE 5—It is good practice to turn the densitometer on before the stain is applied to the filter paper.

NOTE 6—The procurement of five readings of each spot provides an averaging of spot irregularities, while the checking of a minimum of five separate sets of filter paper sheets minimizes the effect of sheet-to-sheet variations.

7.7 Take three densitometer readings on the unstained portion of each disk and average to obtain a blank reading for each disk of filter paper.

7.8 Measure the stained area of each level with the compensating planimeter.

8. Calculation

8.1 Calculate the corrected densitometer readings of Level one, M_1, as follows:

$$M_1 = K_2 - L_1 \quad (1)$$

where:

K_1 = averaged densitometer readings of the colored areas of Level one, and

L_1 = averaged densitometer readings of the unstained areas of Level one.

8.2 Calculate the corrected density times area at Level one, J_1, as follows:

$$J_1 = M_1 \times N_1 \quad (2)$$

where:

N_1 = average area of stained spots of Level one, cm^2.

8.3 Calculate in a similar manner the corrected density times area of the other four levels, J_2, J_3, J_4, and J_5, and obtain the total density times area, J_a, as follows:

$$J_a = J_1 + J_2 + J_3 + J_4 + J_5 \quad (3)$$

8.4 Obtain the percentage distribution as follows:

Percentage distribution, Level one = $(J_1/ J_a) \times 100$

Percentage distribution, Level two = $(J_2/J_a) \times 100$

Percentage distribution, Level three = $(J_3/J_a) \times 100$

Percentage distribution, Level four = $(J_4/J_a) \times 100$

Percentage distribution, Level five = $(J_5/J_a) \times 100$

NOTE 7—Standard "analysis of variance" techniques can be applied to the resultant duplicated two-way classification to separate variance due to spot-to-spot and sheet-to-sheet differences from those of distribution between levels and between formulas.

9. Report

9.1 The report shall include the following:

9.1.1 Identification of the adhesive tested, including type, source, form, antipenetration agent incorporated, if any, and amount,

9.1.2 Amount of adhesive used for each individual test,

9.1.3 Distance of fall of hammer,

9.1.4 Number of tests made for each variable, and

9.1.5 Maximum, minimum, and average values for penetration.

10. Precision and Bias

10.1 At the present time there is no statement of precision and bias concerning the reproducibility of results among laboratories.

11. Keywords

11.1 adhesives; adhesives penetration