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Designation: D 1146 – 00

Standard Test Method for Blocking Point of Potentially Adhesive Layers¹

This standard is issued under the fixed designation D 1146; 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.

This standard has been approved for use by agencies of the Department of Defense.

^{ε1} ~~NOTE—Section 12 was added editorially in December 1994.~~

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. Current edition approved Aug. 26, 1988; March 10, 2000. Published October 1988; May 2000. Originally published as D 1146 – 51 T. Last previous edition D 1146 – 5388 (1984)^{ε1}.

1. Scope

1.1 This test method covers the determination of the blocking point of a thermoplastic or a hygroscopic layer or coating of potentially adhesive material. Potentially adhesive materials comprise those materials in a substantially nonadhesive state which may be activated to an adhesive state by application of heat or solvents.

1.2 The values stated in SI 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. Referenced Documents

2.1 ASTM Standards:

D 907 Terminology of Adhesives²

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adhesive blocking*—the blocking of a potentially adhesive face and a standard test paper.

3.2.2 *blocking*—the adhesion between touching layers of similar or dissimilar material, such as occurs under moderate pressures during storage or use.

3.2.3 *cohesive blocking*—the blocking of two similar, potentially adhesive faces.

3.2.4 *critical humidity*—the lowest humidity at which blocking of a given degree occurs.

3.2.5 *critical temperature*—the lowest temperature at which blocking of a given degree occurs.

3.2.6 *first degree blocking*—an adherence between the surfaces under test of such degree that when the upper specimen is lifted the lower specimen will cling thereto, but may be parted with no evidence of damage to either surface.

3.2.7 *second degree blocking*—an adherence of such degree that when the surfaces under tests are parted one surface or the other will be found to be damaged.

NOTE 1—It is permissible to designate such other degrees of blocking as may be agreed upon between the manufacturer and the purchaser of the adhesive.

4. Significance and Use

4.1 Since some potentially adhesive materials are both thermoplastic and hygroscopic, this test method provides means for estimating, on the same material, both thermoplastic and hygroscopic blocking. Because some requirements are more strict than others, two varying degrees of blocking are recognized and defined: (1) First degree blocking, and (2) Second degree blocking. Two types of blocking are covered: (1) Cohesive blocking, and (2) Adhesive blocking.

5. Apparatus and Materials

5.1 *Constant-Temperature Oven*, capable of maintaining temperatures up to ~~85°C within ±1°C~~, 85 ± 1°C (185 ± 2°F), and of

² Annual Book of ASTM Standards, Vol 15.06.

sufficient size to hold one or more desiccators of the type described in 5.2.

5.2 *Desiccators*, for use as humidity chambers, with a minimum diameter of 150 mm (6 in.). The desiccators are made of glass, with ground flanges and fitting covers. Ground edges are clean and well lubricated with stopcock grease.

5.3 *Weights*, of 0.45 kg (1 lb) each, having a flat base ~~1 in.~~ $645 \text{ mm}^2 (1 \text{ in.}^2)$ in area.

5.4 *Glass Plates*, of plate glass, measuring 38 by 38 mm (~~1½ by 1½ in. (38 by 38 mm)-in.~~).

5.5 *Standard Paper*—General usage filter paper cut in ~~1½ by 38 by 38-mm (1½ by 1½ in. (38 by 38 mm)-in.)~~ squares.

NOTE 2—In cases where standard paper is deemed inadequate, a different paper, or material other than paper, may be substituted by agreement between the manufacturer and the purchaser of the adhesive.

6. Materials

6.1 *Various Salts*, for maintaining constant humidity conditions within the desiccators, as follows:

6.1.1 Anhydrous calcium chloride (CaCl_2), for low humidity, and

6.1.2 The following salts, employed in saturated solution:

Salt	Approximate Relative Humidity over Saturated Solution at 38°C, %
Salt	Approximate Relative Humidity over Saturated Solution at 38°C (100°F), %
Sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O)	50 ^A
Sodium bromide (NaBr)	54 ^A
Sodium nitrite (NaNO ₂)	62 ^A
Sodium acetate (NaC ₂ H ₃ O ₂ ·3H ₂ O)	68 ^A
Sodium chloride (NaCl)	75 ^A
Potassium chloride (KCl)	83 ^B
Ammonium monophosphate (NH ₄ H ₂ PO ₄)	91 ^A

^A "Equilibrium Relative Humidities Above Saturated Salt Solutions at Various Temperatures—Report No. 40 to the American Paper and Pulp Association," Institute of Paper Chemistry, February 15, 1945.

^B "International Critical Tables," Vol 1, 1926, pp. 67–68.

NOTE 3—Desiccators made up with salt solutions are prepared and held at the designated temperatures a sufficient time in advance of use to ensure humidity equilibrium within the desiccators. An excess of salt is maintained throughout the period of testing.

7. Test Specimens

7.1 Cut eighteen test specimens 38 mm (1½-in. (38 mm) in.) square from suitable large specimens for each series of tests (six specimens for cohesive tests and three specimens for adhesive tests in both thermoplastic and hygroscopic blocking).

7.2 Prepare adhesive specimens to conform closely with pertinent commercial practice in respect to adhesive thickness, backing material, and method of preparation.

8. Conditioning

8.1 Condition test specimens and standard paper for determining thermoplastic blocking in a desiccator over anhydrous calcium chloride at $38 \pm 1^\circ\text{C}$ ($100 \pm 2^\circ\text{F}$) for 24 h.

8.2 Condition test specimens and standard paper for determining hygroscopic blocking at the required humidity for 24 h at $38 \pm 1^\circ\text{C}$ ($100 \pm 2^\circ\text{F}$).

9. Procedure

9.1 For cohesive blocking, place two conditioned test specimens, adhesive face to adhesive face, on a glass plate. For adhesive blocking, superimpose three squares of standard paper on the adhesive face of a conditioned test specimen, and place on a glass plate. Align the test specimens, standard paper, and glass plate, so that the edges are flush. Place a 0.45-kg (1-lb) weight (5.3) on top of the aligned specimens, in the center of the ~~1½-in. (38 mm)~~ 38-mm (1½-in.) square area.

9.2 Make triplicate assemblies for each of the following types of test:

9.2.1 *Cohesive Blocking:*

(1) Thermoplastic (2) Hygroscopic.

9.2.2 *Adhesive Blocking:*

(1) Thermoplastic (2) Hygroscopic.

9.3 For thermoplastic blocking, place the above test assemblies immediately in a desiccator over anhydrous calcium chloride at $38 \pm 1^\circ\text{C}$ ($100 \pm 2^\circ\text{F}$) for 24 h. For hygroscopic blocking, place the test assemblies immediately in a desiccator at approximately 50 % relative humidity and at $38 \pm 1^\circ\text{C}$ for 24 h.

9.4 Remove the test assemblies, allow to cool to room temperature, and examine promptly, noting the degree of adherence. Classify the degree of blocking as follows: (1) Free, (2) First degree blocking, (3) Second degree blocking, or (4) Any other degrees of blocking agreed upon.

9.5 If the classification is "free," repeat the test with new specimens at successively higher temperatures (in increments of 5°C (10°F)) for thermoplastic blocking, or successively higher humidities (in increments shown with various salts) for hygroscopic blocking, until first or second or other degree blocking is found, or until a suitably high temperature or humidity is reached.

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the adhesive tested, including thickness or weight per unit area, and method of application to supporting carrier, if any,

10.1.2 Any alternative material employed in 5.5,

10.1.3 Type of test used—whether for cohesive or adhesive blocking, thermoplastic or hygroscopic blocking,

10.1.4 Degree of blocking—free, first degree, second degree, or other,

10.1.5 Results of all determinations made, including: for thermoplastic blocking, the critical temperature or (alternatively) the highest temperature employed; for hygroscopic blocking, the critical humidity or (alternatively) the highest humidity employed; any other critical phenomena.

11. Precision and Bias

~~11.1 At the present time there~~

~~11.1 No information is no basis for a statement of presented about either the precision and or bias concerning the reproducibility of results among laboratories. Test Method D 1146 for measuring the blocking point since the test result is nonquantitative.~~

12. Keywords

12.1 adhesive blocking; blocking; blocking point; cohesive blocking; first degree blocking; second degree blocking

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