

## Designation: D 3354 – 96

# Standard Test Method for Blocking Load of Plastic Film by the Parallel Plate Method<sup>1</sup>

This standard is issued under the fixed designation D 3354; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method yields quantitative information regarding the degree of blocking (unwanted adhesion) existing between layers of plastic film. It is not intended to measure susceptibility to blocking.

1.2 By this procedure, the film-to-film adhesion is expressed as a blocking load in grams which will cause two layers of polyethylene film to separate with an area of contact of 100  $\text{cm}^2$ . The test method is limited to a maximum load of 200 g. See also Test Method D 1893.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 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.

NOTE 1—This test method resembles ISO 11502 in title only. The content is significantly different.

#### 2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics<sup>2</sup>

D 1893 Test Method for Blocking of Plastic Film<sup>3</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

2.2 ISO Standard:

ISO 11502 Determination of Blocking Resistance<sup>5</sup>

#### 3. Terminology

3.1 *Definitions:* For definitions related to plastics, see Terminology D 883.

This edition was revised to meet current ASTM format.

#### 4. Summary of Test Method

4.1 The load in grams required to separate a specimen of blocked film is measured by a balance-beam system similar to an analytical balance. One sheet of a blocked specimen is secured to an aluminum block suspended from the end of the balance beam and the other sheet is secured to an aluminum block fastened to the balance base. Weight is added equivalent to  $90 \pm 10$  g/m to the other side of the beam, until the two films just totally separate, or until they reach 1.905-cm separation.

#### 5. Significance and Use

5.1 Blocking develops in film processing and storage. In most cases the adhesion occurs when touching layers of smooth film are in intimate contact with nearly complete exclusion of air. Adhesion of the touching surfaces is induced by temperature or pressure, or both.

5.2 The procedure of this test method closely simulates the operation of separating film in some end-use applications.

#### 6. Apparatus

#### 6.1 Balance Modification:

6.1.1 A system found satisfactory in a round robin was to modify a heavy-duty, two-pan analytical balance sensitive to 0.1 g by replacing one pan with an aluminum block suspended over another aluminum block attached to the balance base.<sup>6</sup> The essential features of the modification are pictured in Fig. 1. The hook, rod, and universal joint suspension have a total length that will allow the aluminum blocks to mate when the balance is unlocked and balanced. The mating faces shall be square and 100 ± 0.1 mm on each edge with a flat and slightly knurled or sand-blasted finish of root mean square 125.

6.2 *Electro Mechanical Devices*<sup>7</sup>:

6.2.1 Versions of the same test method have been developed which conform to the same test procedure but with different mechanics. The unit adds weight by moving a weight out along a beam at a rate equivalent to the 90-g rate, and instead of weighing the accumulated water, the weight is electronically displayed (Fig. 2).<sup>7</sup>

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>&</sup>lt;sup>3</sup> Discontinued—See 1990 Annual Book of ASTM Standards, Vol 08.01.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

 $<sup>^{\</sup>rm 5}$  Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

<sup>&</sup>lt;sup>6</sup> The analytic balance apparatus is available from Custom Scientific Instruments, Inc., 13 Wing Drive, Cedar Knolls, NJ 07927.

<sup>&</sup>lt;sup>7</sup> The electro mechanical apparatus is available from Kayeness, Inc., East Main St., Honeybrook, PA 19344.

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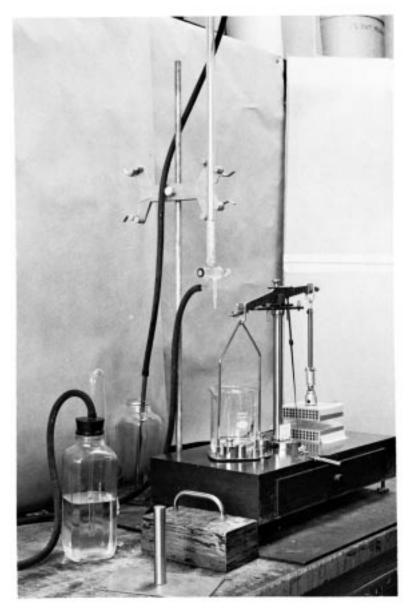


FIG. 1 Balance Modification with Buret Assembly

6.2.2 A typical stationary aluminum block is  $100 \pm 1 \text{ mm} (4 \text{ by 4 in.})$  and  $20 \pm 0.1 \text{ mm}$  thick. If holes are drilled through the blocks for mounting they must be countersunk so that the head of the fastener is below the surface of the block. Back-mounting tapped holes are preferred to leave a smooth surface.

6.2.3 A means must be provided to clamp the two blocks firmly together. However, the clamp must not close with enough force to cause the films to block.

6.3 *Water Supply*—Water is measured from a 100-mL buret into a container resting on the balance pan. The buret should be of the bottom-loading, three-way-stopcock type adjusted to deliver  $90 \pm 10$  mL of water in 1 min (See Fig. 1). Other means such as constant-volume pumps for adding water to this container may be used if the rate of flow is  $90 \pm 10$  mL/min.

6.3.1 An alternate method of adding weight is to move a weight axially along the beam with a precision-drive system.

This may be accomplished using a stepper motor with a digital stepping drive with 60 cycles as its reference control. The weight-addition rate must be equivalent to the 90  $\pm$  10 mL/min.

6.4 Constant Rate of Separation Testing Device—A pair of aluminum blocks 100 by  $100 \pm 1 \text{ mm}$  and  $75 \pm 0.1 \text{ mm}$  thick with appropriate adapters for mounting in a universal testing machine (see Fig. 3).

6.5 Accessory Equipment—Necessary accessories may include a 100 by 180-mm template, double-faced pressuresensitive tape, a stopwatch, and a balance sensitive to 0.1 g.

#### 7. Test Specimens

7.1 Cut block test specimens with a 100 by 180-mm template with the longer length being in the machine direction. Due to variations in gage and blocking tendencies, it is desirable to select several sample locations across the width of



FIG. 2 Electro-Mechanical Device

the film. Five specimens shall be tested. If the test specimens are taken from a roll, care should be taken that the roll is in good condition. The specimens should be taken at least 25 mm below the outer surface of film rolls.

### 8. Conditioning

8.1 Unless otherwise specified, the film to be tested shall be conditioned at least 16 h at  $23 \pm 2^{\circ}$ C and  $50 \pm 5$ % relative humidity. The test samples shall be cut at least 24 h before testing and laid out on a bench where they will not be disturbed until tested. The test should be performed in a controlled atmosphere at  $23\pm 2^{\circ}$ C.

#### **Procedures**

## 9. Procedure A

## 9.1 Modified Balance:

9.1.1 Measure water from a 100-mL buret into a container located on the balance pan. This procedure is preferred when the block force is low and when it is not necessary to refill the buret.

9.1.2 Check the flow of water from the buret. If necessary, adjust the flow to an average of 90  $\pm$  10 mL/min.

9.1.3 Place double-coated tape over two parallel small faces of each aluminum block.

9.1.3.1 A magnet may also be used as a holding means provided it can be demonstrated that no slippage, under maximum load, occurs.

9.1.4 Arrange the balance weights so that the sample side of the block tester is out of balance by about 5 g. Fill the buret with water and allow it to flow into the pan, using no specimen until plates separate. Do not use this first "test tare." Fill the buret with water and determine the average of three "block" determination as "test tare."

9.2 Electro-Mechanical Device:

9.2.1 Place double-coated tape over two parallel small faces of each aluminum block, or place the holding magnets on the blocks as if a sample were being mounted.

9.2.2 Operate the instrument several times, then determine the average of three "block" determinations at "test tare."

9.2.3 Gently place the unseparated film specimen between the two plates with approximately 38 mm of film protruding from each end of the plate. Place both plates in contact with the film aligned with each other. Clamp the plates together with the positioning clamp on the side or lock release on balance. When the plates are brought together, do not slide them sideways because this could possibly break the block.

9.2.4 Carefully separate the edges of the film and stick both protruding edges of the top film to the top plate and the bottom film edges to the bottom plate using the double-faced, pressure-sensitive tape, or holding magnets.

9.2.5 When the film is in place, gently remove the positioning clamp and unlock the release. On the balance start the flow of water through the buret to the water receptacle. Stop the water flow when the films separate. On the electro-mechanical device push the "on" switch. It will automatically stop when the films separate or reach 19.05- mm separation.

9.2.6 Determine the separation load.

9.2.6.1 Record the millilitres of water removed from the buret minus "test tare." Multiply millilitres by 0.9975 to obtain grams of load required to separate the blocked specimen. Record the average load required to separate five blocked film specimens as the blocking load of the sample.

9.2.6.2 With the newer electronic versions, test tare is obtained simply by cycling the unit. The rate is read out on the digital display. This tare must be subtracted from the total displayed load in each test.

#### 10. Procedure B

10.1 Using a wide knife blade or spatula, lift the film specimen and transfer it carefully to a symmetrical position on the lower block with its long ends overlapping the block equally on each side.

10.2 Raise the crosshead of the testing machine until the upper block is resting on the film specimen and is directly above the lower plate.

10.3 Carefully separate the ends of the double layer of film using the sharp edge of the knife blade, and peel back to the edges of the blocks on each side.

10.4 Tape the two ends of the upper layer of film to the upper block, and the two ends of the lower layer of the film to the lower block. Extend the tape an inch around the corners to the front and rear of the blocks so that the ends of the films are securely fastened to the appropriate blocks. Do not otherwise disturb the film or plates during this operation.

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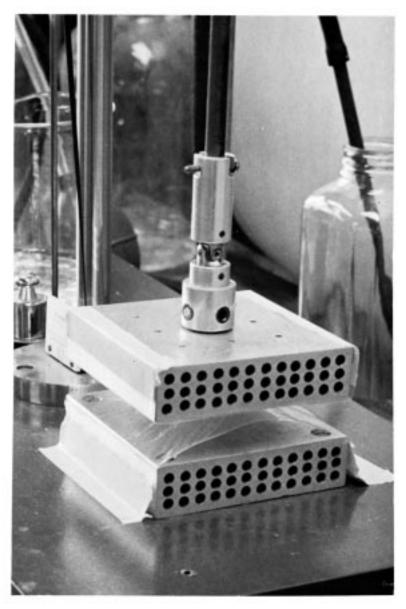


FIG. 3 Suspended and Stationary Aluminum Blocks

10.5 Zero and calibrate the tester according to the instructions provided by the manufacturer so that the full-scale reading on the recorder chart is 200 g.

10.6 Adjust the chart speed to 50.8 mm per min, and the crosshead speed to 5.08 mm per min.

10.7 Start the chart and crosshead travel and allow the testing machine to record the forces required to completely separate the two layers of film. As the test proceeds, the forces acting on the films will vary as the two films arch out, away from the blocks, and peel apart.

10.8 Record the average load (in grams) indicated on the recorder chart and indicate whether the long dimension of the film specimen tested was parallel to the machine direction (MD) or transverse direction (TD).

10.9 Measure and record the thicknesses of both the upper and lower layers of the film after they have been removed from the testing blocks.

#### 11. Report

11.1 Report the following information:

11.1.1 Average load in grams of five test specimens as the blocking load of the sample,

11.1.2 Complete identification of film tested, including type, source, film thickness, process of manufacture, age, and any other pertinent facts,

11.1.3 Date of test, and

11.1.4 Conditioning and testing environment, if different from those recommended in Section 8.

## 12. Precision and Bias<sup>8</sup>

12.1 Precision—Table 1 is based on round robins conducted in 1986 and 1987 in accordance with Practice E 691, involving six materials tested by six laboratories. For each material, all samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of five individual determinations. Each laboratory obtained two test results for each material.

NOTE 2—Caution: The explanations of r and R (12.2-12.2.3) are only

TABLE 1 Blocking

			-		
Material	Average	$S_r^A$	$S_R^B$	r <sup>C</sup>	$R^{D}$
4	7.89	0.60	1.36	1.69	3.64
6	11.5	1.75	2.27	4.97	6.43
1	30.0	3.30	5.08	9.35	14.4
5	47.3	5.70	5.70	16.1	16.1
3	71.2	3.89	6.89	11.0	19.5
2	148	2.36	13.4	6.69	37.9

<sup>A</sup>  $S_r$  = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test result from all of the participating laboratories:

$$S_r = [[(S_l)^2 + (S_2)^2 \dots + (S_n)^2]/n]^{\frac{1}{2}}$$
(1)  
<sup>B</sup>  $S_R$  = between-laboratories reproducibility, expressed as standard deviation:  
 $S_R = [S_r^2 + S_l^2]^{\frac{1}{2}}$ (2)

$$S_{R} = [S_{r}^{2} + S_{L}^{2}]^{\frac{1}{2}}$$
(2)

where:

= standard deviation of laboratory means.  $S_{l}$ 

<sup>C</sup> r = within-laboratory critical interval between two test results =  $2.8 \times S_{c}$ . <sup>D</sup> R = between-laboratories critical interval between two test results =  $2.8 \times S_R$  intended to present a meaningful way of considering the approximate precision of this test method. The data presented in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 12.2-12.2.3 would then be valid for such data.

12.2 Concept of r and R in Table 1—If  $S_r$  and  $S_R$  have been calculated from a large enough body of data, and for test results that were averages from testing five specimens for each test result, then:

12.2.1 Repeatability, r, is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory. Two test results shall be judged not equivalent if they differ by more than the *r* value for that material.

12.2.2 Reproducibility, R, is the interval representing the critical difference between two test results for the material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. Two test results shall be judged not equivalent if they differ by more than the *R* value for that material.

12.2.3 Any judgment in accordance with 12.2.1 or 12.2.2 would have an approximate 95 % (0.95) probability of being correct.

12.3 Bias-There are no recognized standards by which to estimate the bias of this test method.

#### 13. Keywords

13.1 blocking; plastics

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<sup>&</sup>lt;sup>8</sup> Supporting data are available from ASTM Headquarters. Request RR: D20-1041 and 1143.