Standard Test Method for Flex Durability of Flexible Barrier Materials

This standard is issued under the fixed designation F 392; 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 (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the flex resistance of flexible barrier materials. Pinhole formation is the criterion presented for measuring failure, but other tests such as gas-transmission rates can be used in place of the pinhole test.

1.2 This standard does not purport to address all of the safety problems, 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. Specific precautionary statements are given in 5.7.

2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing
D 722 Test Method for Grease Resistance of Paper
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 Specimens of flexible materials are flexed at standard atmospheric conditions (23°C and 50 % relative humidity), unless otherwise specified. Flexing conditions and number and severity of flexing strokes vary with the type of structure being tested. The flexing action consists of a twisting motion followed, in most cases, by a horizontal motion, thus, repeatedly twisting and crushing the film. The frequency is at a rate of 45 cpm.

3.2 Flex failure is determined by measuring the pinholes formed in the structure. These pinholes are determined by using colored turpentine and allowing it to stain through the holes onto a white backing. In addition, other failure criteria such as gas permeation or moisture-vapor transmission can be used at the discretion of the tester.

3.3 The various test conditions are summarized as follows:

3.3.1 Condition A — Full flex for 1 h (that is, 2700 cycles).
3.3.2 Condition B — Full flex for 20 min (that is, 900 cycles).
3.3.3 Condition C — Full flex for 6 min (that is, 270 cycles).
3.3.4 Condition D — Full flex for 20 cycles.
3.3.5 Condition E — Partial flex only for 20 cycles.

4. Significance and Use

4.1 This test method is valuable in determining the resistance of flexible-packaging materials to flex-formed pinhole failures.

4.2 This test method does not measure any abrasion component relating to flex failure.

4.3 Physical holes completely through the structure are the only failures measured by the colored-turpentine-pinhole portion of this test. Failures in the integrity of one of the plies of a multi-ply structure will not be determined by the colored-turpentine test. Gas permeation or moisture vapor transmission tests, or both, can be used in conjunction with the flex test to measure the loss of ply integrity. However, any permeation test requiring a pressure differential will not measure the permeation coefficient in the presence of pinholes.

4.3.1 The various conditions described in this procedure are to prevent testing a structure under conditions that either give too many holes to effectively count and be significant (normally greater than 50), or too few to be significant (normally less than five per sample).

4.4 Measurements on nylon film, possibly because of its hydrophilic nature, have not shown good reproducibility (between laboratories), although the repeatability of the data within a laboratory was good.

5. Apparatus and Reagent

5.1 Flex Tester, designed so that it can be set up in accordance with the specifications listed in Section 8. This apparatus shall consist essentially of a 90-mm (3.5-in.) diameter stationary mandrel and a 90-mm diameter movable mandrel spaced at a distance of 180 mm (7 in.) apart from face-to-face at the starting position (that is, maximum distance)

1 This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods.


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.


4 The Gelbo Tester, which is capable of producing the prescribed flexing action, available from the United States Testing Co., Inc., 1415 Park Ave., Hoboken, NJ 07030, or its equivalent, has been found satisfactory for this test method.
of the stroke. Mandrels shall contain vents to prevent pressur-
ization of samples. The specimen supporting shoulders on the
mandrels shall be 13 mm (0.5 in.) wide. The motion of the
movable mandrel is controlled by a grooved shaft to which it
is attached. For the full or maximum stroke the groove is
designed to give a twisting motion of 440° in the first 90 mm
of the stroke of the movable mandrel, followed by a straight
horizontal motion of 65 mm (2.5 in.), so that at the closed
position the mandrels are 25 mm (1 in.) apart. The motion of
the machine is reciprocal with a full cycle consisting of the
forward and return strokes. The machine operates at 45 cpm.

5.1.1 Fig. 1 shows the planar evolution of the helical groove
in the driven shaft to give the required 440° (37° helix angle)
twisting motion and the straight horizontal motion.

5.1.2 For the partial flex used with Condition E the movable
head is set to travel only 80 mm (3.25 in.) of the 180-mm
(7-in.) spacing. Therefore, only approximately 90% of the
twisting stroke is utilized giving a twisting motion of only
400°, and none of the horizontal stroke is utilized.

5.2 Tape, flexible, double-sided, pressure-sensitive, not
more than 13 mm (0.5 in.) wide.

5.3 Template, for cutting 200 by 280-mm (8 by 11-in.)
samples.

5.4 Paint Brush, large, 50 to 150 mm wide.

5.5 Tissue Paper, absorbent.

5.6 White Paper, such as uncoated bond paper, at least as
large as the film samples.

5.7 Turpentine (Colored, Water-Free)—To 100 mL of pure
gum spirits of turpentine (chemically pure grade, sp gr 0.860 to
0.875 at 15°C) add 5 g of anhydrous calcium chloride (CaCl₂)
and 1.0 g of oil-soluble red dye. Stopper the container, shake
well, and let stand for at least 10 h, shaking occasionally. Then
filter through a dry filter paper at a temperature of approxi-
mately 21°C, and store in an airtight bottle.

NOTE 1—Caution: Use of these materials requires that appropriate
safeguards be used to avoid hazards of skin contact, inhalation, and
flammability.

6. Test Specimens—All Conditions

6.1 Cut the samples into 200 by 280-mm (8 by 11-in.) flat
sheets with the 200-mm dimension in the direction to be tested.
This will also be in the direction of the flex-tester axis.

6.2 Flex test four specimens in their machine direction and
four in their transverse direction. In addition, test a control set
of four, adjacent, unflexed specimens (either direction) for
pinholes.

6.3 Do not seal or tape the sides of the specimens, but leave
them open. Use double-sided pressure-sensitive tape, not more
than 13 mm (0.5 in.) wide, to attach the unsealed specimen in
the shape of a cylinder to the flex-tester mandrels.

7. Conditioning

7.1 Condition the specimens for at least 24 h at 50 ± 5%
relative humidity and 23 ± 2°C, unless otherwise specified as

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![FIG. 1 Planar Evolution of Gelbo Shaft Helical Groove (31.8-mm (1.25-in.) Diameter Shaft)](image-url)
agreed upon between the purchaser and the seller. (See Practice D 618 for other standard atmospheric conditions.)

8. Procedure

8.1 Flexing:

8.1.1 Atmospheric Conditions—Conduct flexing at the standard atmospheric conditions of 23°C and 50% relative humidity, unless otherwise specified.

8.1.2 Condition A:

8.1.2.1 Flex Tester Setup—Setup the flex tester for maximum throw and twisting action. This setup gives a twisting motion of 440° in the first 90 mm (3.5 in.) of stroke, and is followed by a straight horizontal motion of 65 mm (2.5 in.) at 45 cpm. With this setup, space the face of the moving mandrel at a distance of 180 mm (7 in.) from the face of the stationary mandrel when the moving mandrel is at its starting position. In the closest position the moving mandrel is 25 mm (1 in.) from the stationary mandrel.

8.1.2.2 Flexing—Attach the flexible barrier specimen to the flex-tester mandrels, which have been lined with double-sided pressure-sensitive tape. Turn the flex tester on, and allow the specimen to flex for 1 h at 45 cpm (that is, 2700 cycles).

8.1.3 Condition B—Test conditions are the same as Condition A, except that the flex period is 20 min at 45 cpm (that is, 900 cycles at full flex and twisting action).

8.1.4 Condition C—Test conditions are the same as Condition A, except that the flex period is 6 min at 45 cpm (that is, 270 cycles at full flex and twisting action).

8.1.5 Condition D—Test conditions are the same as Condition A, except that the flex period is 20 cycles at 45 cpm (that is, 20 cycles at full flex and twisting action).

8.1.6 Condition E—Setup the flex tester for the partial flex described in 5.1.2. Here the movable head is set to travel only 80 mm (3.25 in.) of the 180-mm (7-in.) spacing (the distance between the mandrels at their maximum separation or starting position). Therefore, only about 90% of the twisting stroke giving a twisting motion of only 400° is utilized and none of the horizontal stroke is utilized. When the mandrels are at their closest position they will be 95 mm (3.75 in.) apart. The partial flex period under this “short stroke” setup will be 20 cycles at 45 cpm.

8.2 Pinhole Count:

8.2.1 Remove the flexible barrier specimen from the flex tester and mark the 150 by 200-mm (6 by 8-in.) center area of white paper corresponding with the center area of the specimen with the 150-mm (6-in.) dimension in the flex tester-axis direction.

8.2.2 Tape the specimen to the sheet of white paper against a flat surface.

8.2.3 Paint the specimen with colored turpentine solution using multiple strokes of the brush and allow to set for 1 min.

8.2.4 After 1 min, wipe the colored turpentine off with absorbent tissue paper, pressing the specimen against the white backing while wiping.

8.2.5 Remove the specimen from the paper and count each strike-through on the paper as a pinhole. Where color patterns overlap, but distinct center marks are seen, count as separate pinholes. Count all strike-throughs regardless of size, even if they are pin-point size, as pinholes. Count only the pinholes in the 150 by 200-mm (6 by 8-in.) area.

9. Report

9.1 The report shall include the following:

9.1.1 Individual values and average pinholes per 300 cm² (48 in.²) on quadruplicate determinations in machine direction and separately in transverse direction,

9.1.2 Any unusual failures such as tears,

9.1.3 On each sample under test measure the pinholes of the four unflexed specimens using procedure of Section 8 (this will determine if some pinholes are already in the film that are not the result of flexing). Report the individual values and average of these data as “Pinholes on Unflexed Control.”

9.1.4 Test method used, including the number of cycles and whether full or partial flex was used.

9.1.5 Sample conditioning and test conditions used, and

9.1.6 Where applicable, sample thickness and structure.

10. Precision and Bias

10.1 Precision—Data presented in Table 1 is based on a round-robin series of tests conducted in 1992 in accordance with Practice E 691, involving four materials tested by five laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared and tested at the laboratory that provided the results. Each test result is based upon quadruplicate determinations in the machine direction of the material. Each laboratory obtained two replicates for each material.

10.2 Bias—There are no recognized standards by which to estimate bias of this test method.

11. Keywords

11.1 barrier; barrier materials; flex crack; flex resistance; flexible; pinhole

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5 Supporting data are available from ASTM Headquarters. Request RR:F02-1011.

### TABLE 1 Pinhole Data

Note: 1—Values expressed in pinholes/300 cm² (48 in.²).

<table>
<thead>
<tr>
<th>Material Description</th>
<th>Test Condition</th>
<th>Average s_r ²</th>
<th>s_0 ²</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8 Mil PVdC ctd Cellophane</td>
<td>D</td>
<td>3.45</td>
<td>1.47</td>
</tr>
<tr>
<td>0.92 Mil Oriented Polyester</td>
<td>C</td>
<td>3.18</td>
<td>0.87</td>
</tr>
<tr>
<td>0.48 Mil Oriented Polyester</td>
<td>C</td>
<td>0.18</td>
<td>0.24</td>
</tr>
<tr>
<td>1.5 Mil Low-Density Polyethylene</td>
<td>C</td>
<td>8.15</td>
<td>1.24</td>
</tr>
</tbody>
</table>

^A s_r = within-laboratory deviation.

^B s_0 = between-laboratory deviation.