Designation: F119 – 82 (Reapproved 2015)

Standard Test Method for Rate of Grease Penetration of Flexible Barrier Materials (Rapid Method)[1]

This standard is issued under the fixed designation F119; 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 U.S. Department of Defense.

1. Scope

1.1 This test method provides standard conditions for determining the rate of grease penetration of flexible barrier materials. Pinholes, which can be measured by a separate test, will increase the rate of grease penetration as determined by this test method.

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

1.3 *This standard does not purport to address the safety concerns 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]

D374 Test Methods for Thickness of Solid Electrical Insulation (Withdrawn 2013)[3]

D618 Practice for Conditioning Plastics for Testing

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

2.2 *TAPPI Standard:*

TAPPI T465 sm-52 Creasing of Paper for Water Vapor Permeability Tests[4]

3. Summary of Test Method

3.1 Flexible barrier materials, uncreased or creased by a standard procedure, are exposed on one side to grease contained in a weighted cotton patch. The time required to show a visual change caused by wetting (reduction in light scattering) of a ground-glass back-up plate is measured.

4. Significance and Use

4.1 This test method is valuable in the development and selection of flexible barrier materials suited for use as grease barriers.

4.2 The test is rapid in comparison with other methods because of the extremely small quantity of oil required for detection (about 6 µg). The actual time to failure is a multiple of the values obtained by this test method. When permeation is through an absorbent structure such as kraft paper coated with polyethylene, the failure times will be longer and variable, depending on the variation in porosity and thickness of the structure.

5. Apparatus

5.1 *Backing Plates,* ground-glass, 50 by 50 by 3-mm (2 by 2 by ⅛-in.), very fine grind on one side only.

NOTE 1—The ground-glass backing plates may be prepared from 2 by 2-in. squares of ⅛-in. plate glass by lightly grinding the surface with a silicon carbide abrasive as follows: Place a piece of plate glass approximately 1ft square on a flat surface. Mix some abrasive and water in small quantities to make a fairly uniform paste. Place one of the 2-in. squares of plate glass face down in the abrasive paste, and rotate it in a figure eight movement with the finger tips using the slightest amount of pressure. When the paste gets too heavy, remove the plate glass square from the bed and wash it to remove all traces of abrasive. Add more water to the thick abrasive paste on the bed. (If necessary, add small amounts of abrasive.) Continue the process until a uniform and light overall etch is present on the face of the square. When a uniform etch has been obtained, wash the square thoroughly to remove all traces of abrasive and pat dry with lens tissue.

5.2 *Weights,* 50-g, 20 mm (0.75 in.) in diameter at the base.

5.3 *Patches,* rifle cleaning, cotton flannel.

5.4 *Medicine dropper.*

5.5 *Forced-Circulation Oven,* designed to maintain a test temperature of 40 or 60°C within ±1°C.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and is the direct responsibility of Subcommittee F02.10 on Permeation.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

5.6 *Creasing Surface,* consisting of a flat rectangular plate (for example, a piece of machined metal plate about 10 mm thick or a piece of plate glass) with a width at least 75 mm (3 in.) on all sides.

5.7 *Creasing Platen,* consisting of a 5.5-kg (12-lb) square metal bar with 65-mm (2.5-in.) sides and a flat base.

NOTE 2—The developers of this method believe that a standard crease is easier to obtain with a flat platen than with a roller. The weight of the platen was selected to conform to the weight loading per inch of crease in TAPPI T465 sm-52.

5.8 *Flat Strip* of wood or metal to give the specimen a preliminary light crease.

5.9 *Thickness Measuring Device,* capable of measuring thickness of specimens in compliance with Test Methods D374.

6. Reagents

6.1 *Animal Oil* (lard).

6.2 *Mineral Oil.*

6.3 *Vegetable Oil.*

6.4 Other reagents, such as butter, tallow, and oils. These may be substituted for standard reagents listed above to simulate actual end-use conditions. Standard reagents are, however, recommended for purposes of interlaboratory comparisons.

7. Sampling, Test Specimens, and Test Unit

7.1 No single procedure for sampling is adequate for all situations. Sampling, however, should be designed to provide the desired result in each situation. Therefore, Practice D1898 is recommended as a guide in designing appropriate sampling procedures for the purposes at hand.

7.2 *Flat Specimen*—Cut a minimum of three test specimens 60 by 60 mm (2⅜ by 2⅜ in.) from each sample after conditioning at least 40 h at 23 ± 2°C and 50 ± 5 % relative humidity. Measure the thickness of each specimen in five equally spaced positions as described in Test Methods D374 to three significant figures, or to 0.00025 mm (0.01 mil) when three significant digits cannot be obtained. Record the measurements.

7.3 *Crease Test*—Prepare a minimum of six test specimens as described in 7.2. Fold three specimens with top side up and three specimens with bottom side up. Fold through the center parallel to one side and press a flat strip of wood or metal down on the fold to give the specimen a preliminary light crease. Tilt the 5.5-kg (12-lb) creasing platen and slip the folded specimen under it and parallel to its edge, until the fold lies at the center of gravity of the platen. Gently lower the platen and let it rest on the specimen for 15 s. Remove the specimen and unfold it. Then fold it on the same side to make a new crease at a 90° angle to the first crease, following the same procedure. Flatten the specimen by unfolding it and placing it under platen for 15 s.

7.4 *Test Unit*—A minimum of three similar specimens make up a test unit.

8. Procedure

8.1 Place the test specimen of measured thickness on a clean ground-glass backing plate. The film specimen should more than cover the glass backing plate so premature edge creep failure does not occur. Test specimens should include flat, creased into, and creased away from the side that will come into contact with grease in the package.

NOTE 3—Previously used ground-glass backing plates should be thoroughly cleaned by allowing them to stand overnight in a chromic acid solution.

8.2 Cut the cotton flannel rifle cleaning patches into 20-mm (0.75-in.) diameter disks to just fit under the 50-g weight.

8.3 Place two cotton flannel disks on top of each other at the center of the test specimen.

8.4 Place the 50-g weight on the patches and preheat the entire assembly (glass, test specimen, cotton disks, and weight) to the desired test temperature (40 or 60 ±1°C) for 30 min. This is particularly necessary when short failure times are expected, where ±30 min would be critical.

8.5 With the assembly still in the oven, remove the weights and add six drops of reagent, oil, or grease, to the cotton disks. If necessary, melt the grease so it can be added dropwise. Periodic addition may be required when volatile reagents are used under the option of 6.4.

8.6 Replace the 50-g weights on the oiled patches.

8.7 Close the oven door and note the time.

8.8 At periodic intervals, depending on the length of time to anticipated failure (that is, every 15 min for the first hour; every 30 min for the next 4 h; then at convenient intervals), lift the test specimen, cotton disk, and weight from the ground-glass plate as a unit and observe the surface of the glass against a dark background. Record the time at which the first trace of wetting (reduction in light scattering) is visible at the position of the weight. If no failure is visible, replace the assembly in the oven.

8.9 If failure as indicated by the first visible wetting occurs in less than 1 h at elevated temperatures, the disruption in temperature by the act of observing the sample is sufficient to alter the end point significantly. In these cases repeat the test and check the end point at different times for each of several tests until failure time is obtained.

9. Calculation

9.1 Calculate the average and standard deviation of the thickness measurements from each specimen and record them.

10. Report

10.1 The report shall include the following information:

10.1.1 Complete sample identification.

10.1.2 Type specimen used, that is, flat or creased, and the size of the test unit.

10.1.3 Type of reagent used.

10.1.4 Test temperature.

10.1.5 Thickness, average, and standard deviation for each specimen tested.

10.1.6 Average time to failure plus the minimum and maximum time to failure for each sample with the samples nominal thickness.

11. Precision

11.1 The data presented here were calculated from the results of a round robin participated in by five laboratories.[5] The test covered four substrates: 1 and 2 mil low-density polyethylene (LDPE) film; 0.75 mil polypropylene homopolymer film; 1 mil low-density polyethylene on 40-lb kraft paper. Oils tested were mineral oil and vegetable oil. Substrates were tested flat, creased into oil and creased away from oil. The test temperature was 60°C.

11.2 The results of the round robin showed an overall one sigma standard deviation of 31 % for uncreased samples. No significant differences in the average results were observed on creasing the samples before testing. This is an excellent agreement considering: (*1*) the large dependency of oil permeation on temperature (±1°C test temperature = −14 to + 18 % deviation); (*2*) film thickness (1.25 ± 0.1 mil = ±13 % deviation); (*3*) that this was the first time most of the laboratories had run the test; and (*4*) the test end point is not read continuously, but at intervals. Even so, this permeation test can distinguish between 1 and 2 mils of polyethylene rather than merely rank materials by decade differences.

NOTE 4—The dependence of mineral oil permeation on temperature and film thickness was determined separately by one of the round-robin testing laboratories. The effect of film thickness is obtained from empirical measurements which resulted in the following equation:

$$\frac{P_2}{P_1} = \left(\frac{t_2}{t_1}\right)^{1.635} \quad (1)$$

where:

P = time to failure, and
t = film thickness.

This relationship was obtained on films of two different materials (low-density polyethylene and ionomer) at 23, 40, and 60°C over a range of thicknesses of 0.5 to 4.0 mils.

11.3 *Repeatability (Within-Laboratory)*—Each laboratory made three determinations simultaneously on each substrate and oil combination. At least 82 % of the time each sample in these triplicate determinations gave *identical* values.

11.4 *Reproducibility (Between-Laboratories)*—The data showed an overall standard deviation of 31 % (uncreased samples) from the average values reported by each laboratory. The standard deviations ranged from 19.4 to 46.4 % between laboratories for each substrate and oil tested. No significant change in the average values could be attributed to creasing the particular samples used in this round robin.

11.5 The round-robin data resulted in the following average $(\bar{X})$ and range of permeation times among the testing laboratories:

Substrate		Hours to Failure, 60°C	
		Mineral Oil	Vegetable Oil
LDPE, 1 mil	$\bar{X}$	0.375	1.58
	range	0.25 to 0.5	0.9 to 2.0
LDPE, 2 mil	$\bar{X}$	0.905	2.30
	range	0.4 to 1.5	1.5 to 3.0
Polypropylene, 0.75 mil	$\bar{X}$	17.5	264
	range	14 to 22	168 to 356
LDPE/40-lb Kraft, 1 mil	$\bar{X}$	15.2	38.3
	range	12 to 20	27 to 50

12. Keywords

12.1 flexible barrier packaging; grease penetration

[5] Supporting data describing the work of the Round-Robin Task Group are available from ASTM International Headquarters. Request RR-F02-0001.