Standard Test Method for Detecting Leaks in Nonporous Packaging or Flexible Barrier Materials by Dye Penetration[1]

This standard is issued under the fixed designation F3039; 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 Method A of this test method defines a procedure that will detect and locate a leak equal to or greater than a channel formed by a 50 µm [0.002 in.] wire in the edge seals of a nonporous package. A dye penetrant solution is applied locally to the seal edge to be tested for leaks. After contact with the dye penetrant for a minimum specified time, the package is visually inspected for dye penetration or, preferably, the seal edge is placed against an absorbent surface and the surface inspected for staining from the dye.

1.2 Method B for this test method also defines a procedure that will detect and locate a leak equal to or greater than 10 µm [0.00039 in] diameter in a nonporous flat sheet. The flat sheet is placed on an absorbent surface and then a dye penetrant is spread across the surface of the sheet, preferably using a small roller to apply pressure on the sheet to ensure adequate contact between the absorbent surface and the bottom surface of the sample being tested. The flat sheet is carefully removed and the absorbent surface is inspected for staining from the dye.

1.3 These test methods are used for both transparent and opaque nonporous surfaces.

1.4 These test methods require that the dye penetrant have good contrast to the materials being tested and/or the absorbent surface.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.6 *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:*[2]

E171 Practice for Conditioning and Testing Flexible Barrier Packaging

2.2 *ANSI Standards:*[3]

Z1.4 Sampling Procedures and Tables for Inspection by Attributes

3. Terminology

3.1 *Definitions:*

3.1.1 *dye penetrant*—an aqueous solution of a dye and a surfactant designed to penetrate and indicate a defect.

4. Significance and Use

4.1 Contaminants may enter the package through leaks. Alternatively, product may be lost from the package through leaks. These leaks are frequently found at seals between package components of the same or dissimilar materials.

4.2 Ingress or egress of gas or moisture through leaks in a package can also degrade sensitive contents.

4.3 There is no general agreement concerning the level of leakage that is likely to be deleterious to a particular package. However, since these tests are designed to detect leakage, components that exhibit any indication of leakage may be rejected.

4.4 These procedures are suitable for use to verify and locate leakage sites. They are not quantitative. No indication of leak size can be inferred from the test. Therefore, this method is employed as a go/no-go test.

4.5 These tests are destructive. No package or component test samples exposed to dye penetration testing may be used for final product packaging.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and is the direct responsibility of Subcommittee F02.40 on Package Integrity.

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

5. Apparatus

5.1 Means of breaching one of the packaging materials such as a small knife.

5.2 *Dye Dispenser,* such as an eyedropper or syringe for injection of the dye penetrant solution, or for application of the dye penetrant to the surface of the sheet sample for Method B.

5.3 *Microscope* or optical loop with magnification of 5× to 20× (optional).

5.4 *Absorbent Surface,* such as a white chromatography paper, white filter paper, or white paper towels. Bright white printer or photocopier paper is preferred for the flat sheet (Method B) test.

NOTE 1—An embossed or uneven surface on the absorbent surface can inhibit consistent contact between the bottom surface of the sample and the absorbent surface and can interfere with leak detection.

5.5 Roller or other means to ensure intimate contact of the bottom of the sample being tested with the absorbent surface (Method B only).

5.6 Aqueous dye penetrant solution consisting of a carrier, wetting agent, and indicator dye with a surface tension of 32 dynes/cm or less. One possible dye penetrant solution formulation consists of, by weight:

Wetting agent	Polyethylene glycol octylphenyl ether[A]	3 %
Indicator dye	Toluidine blue[B]	0.05 %
Carrier	Distilled water	96.95 %

[A] Polyethylene glycol octylphenyl ether is sold under the brand name Triton X-100.
[B] The chemical name for Toluidine blue is 3-Amino-7-(dimethylamino)-2-methylphenothiazin-5-ium chloride.

5.6.1 Because of the viscosity of the polyethylene glycol octylphenyl ether, the preparation of the solution is most easily accomplished by first taring a container with about 10 % of the required amount of distilled water on a scale. The appropriate amount of polyethylene glycol octylphenyl ether is added to the distilled water by weight and the mixture stirred or shaken. Once the polyethylene glycol octylphenyl ether is dissolved, the remaining distilled water can then be added, followed by the toluidine blue dye.

5.6.2 The solution must remain homogeneous. If precipitate is noted, the solution must be replaced.

5.6.3 If substitutes for the toluidine blue and/or Triton X-100 are used, their precision and bias must be experimentally determined.

6. Safety Precautions

6.1 Injecting dye penetrant into a package with a hypodermic needle and syringe is a common method for performing the seal channel detection portion of this test. Caution should be taken if this practice is chosen as it can result in puncture of the skin with a contaminated needle. An alternative approach is to dispense the dye penetrant via a flexible tube attached to a syringe through an opening formed with an appropriate cutting instrument.

7. Test Specimen

7.1 The test specimen may consist of a complete packaged product (blemished, rejected, or dummy devices may be used if they will not affect test results and are recorded prior to the test) to assess the package for channels in seals or leaks in the surface of the material. Empty packages, or edge seal samples or sheet material may also be used for testing for channels or leaks in the seals or leaks in the surface of the material.

8. Calibration and Standardization

8.1 Because this test is not quantitative, calibration is not applicable.

9. Sampling

9.1 The number of samples tested should be adequate to be predictive of performance. Caution should be taken when eliminating samples with defects as this can bias results. See ANSI/ASQ Z1.4.

10. Conditioning

10.1 Packaging must be free of condensation or any other source of liquid water. Water already in the channel, hole, or potential leak point may render them undetectable with a dye penetrant. If there is any indication that the package has been exposed to any liquid, it must be thoroughly dried at its typical storage temperature before testing.

10.2 See Practice E171 for conditioning guidance.

11. Procedure

11.1 *Method A*—Testing for channels or leaks in the seal:

11.1.1 Inject dye penetrant into the package or along seal edge at a volume of at least 0.25 ml per 25 mm [1 in.] of seal length.

NOTE 2—If puncturing the packaging to allow injection of the dye penetrant solution, care should be taken not to puncture through or damage other package surfaces. Puncturing of the package is facilitated if it is done adjacent to a dummy product inside the package. The product will provide a tenting effect that will separate the two sides of the package, reducing the chance of accidental puncture of both sides.

11.1.2 Allow the dye penetrant solution to remain in contact with the seal edge for approximately 5 s. Channels will be detected within this time period.

11.1.3 If the package has a transparent side, the seal may be examined visually through this side. An optical device with 5× to 20× magnification may be used for detailed examination. If the package is opaque or if additional verification is desired, the outside edge of the seal being tested should be placed against an absorbent surface for approximately 5 s. After carefully lifting the package, examine the absorbent surface. The presence of stains indicates a channel or leak.

NOTE 3—If the package being tested has unsealed material between the outer edge of the seal and the outer edge of the package (i.e., a "skirt"), fold back the skirt material when placing the edge seal against the absorbent surface.

11.1.4 Rotate the package as necessary to expose each seal edge to the dye penetrant solution and repeat the examination detailed in 11.1.2 and 11.1.3. Inject additional dye as needed to ensure complete edge exposure.

11.2 *Method B*—Testing for holes or leaks in a flat surface:

11.2.1 If testing a package, carefully cut the surface to be inspected so that the packaging material can lay flat.

11.2.2 Place the sample, sheet or cut package, to be examined on an absorbent surface so that it lays flat without puckers or wrinkles. Analyst may use tape or small weights to secure the sheet sample to the adsorbent surface.

11.2.3 Saturate an absorbent pad with dye penetrant solution and wipe across the surface of the test sample or apply dye penetrant solution to the surface of the test sample with an eye dropper or pipette. Dye penetrant solution should contact all areas exhibiting questionable surface anomalies taking care not to allow dye penetrant solution to flow over the edge of the sample.

11.2.4 Once areas of interest of the sample have been wiped with dye penetrant solution, use a small roller or other means to apply pressure to the sample to ensure adequate contact between the absorbent surface and the bottom surface of the sample being tested. Wipe excess dye from sample using a clean absorbent pad and carefully lift the sample.

11.2.5 Examine the absorbent surface circling any stains. The presence of stains indicates a hole or leak.

12. Report

12.1 Report the following information:

12.1.1 Lot number and source of material, date, time, location, and operator of test and complete identification of materials being tested.

12.1.2 Any conditioning of the materials.

12.1.3 Dye penetrant formulation.

12.1.4 Test(s) performed: Method A, Method B, or both.

12.1.5 Method of visual inspection: aided or unaided.

12.1.6 Results:

12.1.6.1 Evidence of dye penetration to the opposite side of the seal via a defined channel shall be taken as the indication of the presence of a leakage site.

12.1.6.2 Evidence of dye penetration to the absorbent surface shall be taken as the indication of the presence of a leakage site.

12.1.6.3 A qualitative description or sketch of the leakage sites.

12.1.7 Any and all deviations from standard.

13. Precision and Bias

13.1 *Method A*—The precision of this test method is based on an interlaboratory study conducted in 2012. Ten laboratories participated in this study. Channel defect samples were created with a 50 µm (0.002 in.) Tungsten wire in a 9.5 mm [0.375 in.] wide seal. Note that none of the samples tested had a skirt. Each group of 50 samples contained 20–30 randomly distributed defect samples, with the balance of the samples being defect-free. Each participant analyzed 200 randomly coded samples (4 groups of 50 samples each). Every analyst reported results to indicate the presence or absence of a channel. The results were summarized in Table 1; the details are given in RR:F02-1034.[4]

13.1.1 The materials tested were identified as:

13.1.1.1 Clear materials.

13.1.1.2 PET/LDPE

13.1.1.3 OPP/sealant

13.1.2 Opaque materials.

13.1.2.1 PET/Al Foil/LDPE

13.1.2.2 PA/Al Foil/EAA

TABLE 1 Summary of Results

Total specimens tested	False positives	False negatives	95% confidence interval	Error rate (%)
2000	2	4	99.13–99.94	0.3

13.2 *Method B*—The precision of this test method is based on an interlaboratory study conducted in 2013/2014. Eight laboratories participated in this study. Pinhole defect samples were created by drilling holes in flat sheet materials with a laser. Each group of 18 samples contained 3 control samples (no pinhole), and 3 samples each at 10 µm, 20 µm, 30 µm, 40 µm, and 50 µm randomly distributed. Four sets of these samples were obtained, and the samples were returned to the study director after the first four labs tested them. The hole sizes of these samples were verified before they were sent out to the second set of four labs. Every analyst reported results to indicate the presence or absence of a pinhole. The results were summarized in Table 2; the details are given in RR:F02-1040.[5]

13.2.1 The material used in the interlaboratory study for Method B was PET/Al Foil/PE.

TABLE 2 Summary of Results [A] **,** [B]

Hole Size, µm	Sample Size	Correct Responses	Incorrect Responses	Probability of Detection	CI Lower Bound	CI Upper bound
0	25	24	1	0.960	0.796	0.999
10	26	20	6	0.769	0.564	0.910
20	23	21	2	0.913	0.720	0.989
30	23	22	1	0.957	0.781	0.999
40	24	24	0	1.000	0.883	. . .
50	23	23	0	1.000	0.878	. . .

[A] Sample size varies because some samples were found to be either blocked or significantly smaller during the hole size verification step and were reclassified.
[B] Lower CI and Upper CI refer to the 95% confidence intervals (CI).

13.3 *Bias*—There is no bias for pass/fail test methods.

14. Keywords

14.1 channel; dye penetrant; flexible packaging; leaks; nonporous; pinhole

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1034. Contact ASTM Customer Service at service@astm.org.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1040. Contact ASTM Customer Service at service@astm.org.

ANNEX

(Mandatory Information)

A1. CAPILLARY ACTION

A1.1 The use of dye penetration to detect pinholes and channels relies on capillary action. Capillary action is the tendency for liquids to spontaneously rise in small tubes against the force of gravity. It occurs when the adhesive forces between the liquid and the surface of the walls of the tube is greater than the cohesive forces between the liquid molecule. Capillary action explains why paper towels absorb liquids and why fluids are transported through plants.

A1.2 A liquid will form an interface with a second liquid or gas. The actual physical chemistry of such interface surfaces is extremely complex. Generally speaking, molecules within the liquid repel each other because of their close packing. In contrast, molecules at the surface are less dense and attract each other. Since these surface molecules only have other liquid molecules on one side, the mechanical effect is that the surface is in tension.[6] The resultant surface tension is measured in dynes/cm and represented by the symbol θ. The surface tension for water at 20 °C [68 °F] is 73 dynes/cm.[6] Water's surface tension can be altered dramatically with the addition of contaminants such as surfactants.

A1.3 When a solid surface intersects with a liquid-gas interface, the angle formed by the liquid is the contact angle θ. If the contact angle is less than 90°, the liquid wets the solid and if it is greater than 90° it is nonwetting. The greater the difference between the surface tension of the liquid and the surface energy of the solid surface, the more the liquid will wet the surface of the solid and the smaller the contact angle θ.

A1.4 The height to which the liquid will rise in a tube is a function of both the surface tension of the liquid and the contact angle formed with the tubes surface. The relationship is represented by the equation[7]:

$$h = \frac{2\gamma\cos\theta}{\rho g r} \quad \text{(A1.1)}$$

where:

γ = liquid-air surface tension,
θ = contact angle,
ρ = density of the liquid,
g = acceleration due to gravity, and
r = radius of the tube.

A1.5 A pinhole or channel is in essence a tube. Because the dye penetrant solution has a surface tension less than the surface energy of the materials generally used for flexible packaging materials (see Table A1.1), it will readily wet the surface of the pinhole or channel. The dye penetrant solution does not need to be "pushed" into the channel or hole. As long as the dye penetrant solution makes contact with the channel or hole, capillary action can occur.

[6] White, Frank M., *Fluid Mechanics*, McGraw-Hill, Inc. 1986.

[7] Batchelor, G. K., *An Introduction To Fluid Dynamics*, Cambridge University Press, 1967.

TABLE A1.1 Surface Energy of Common Packaging Materials (untreated)[A]

Material	Surface Free Energy (dynes/cm)
Aluminum Foil	41.2
Polyamide-6.6 (nylon 6,6)	44.3
Polycaprolactam, aramid 6 (nylon 6)	45.4
Polyethylene (PE)	33.5
Polyethyleneterephthalate (PET)	44.0
Polyisobutylene	33.2
Polypropylene (PP)	30.2
Polystyrene	40.7
Polyvinyl acetate (PVA)	38.5
Polyvinyl alcohol (PVOH)	44.2
Polyvinyl chloride (PVC)	40.1
Polyvinylidene fluoride (Saran)	43.7

[A] http://www.accudynetest.com/polytable_01.html

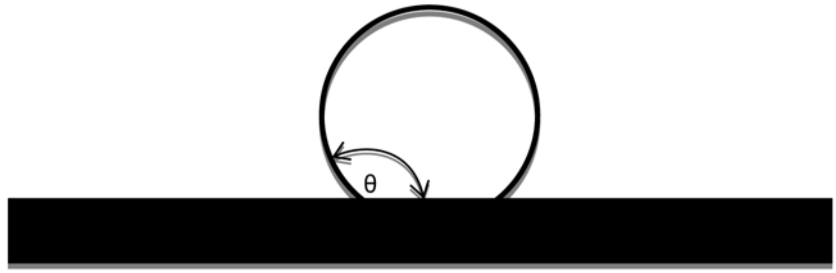

FIG. A1.1

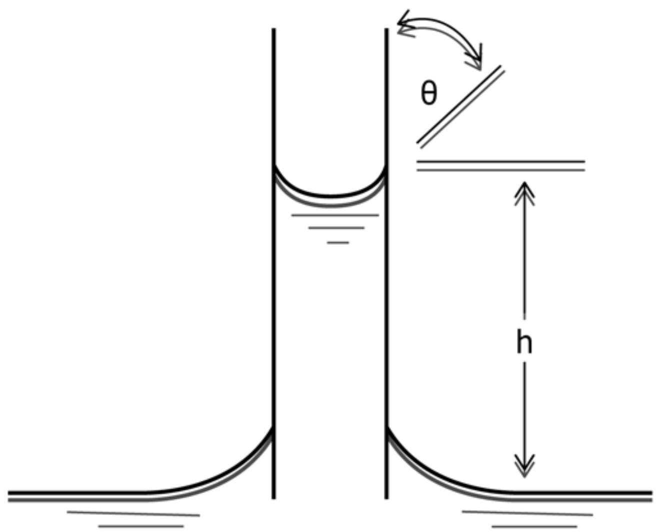

FIG. A1.2