



Designation: F3039 – 23

Standard Test Method for Detecting Leaks in Nonporous Packaging or Flexible Barrier Materials by Dye Penetration¹

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

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1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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, n*—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

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>.

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.

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, a surfactant, and an indicator dye. Ideally, the surfactant chosen should be nonionic. The chemical properties of the surfactant should be as follows: surface tension between 30 mN/m to 33 mN/m, hydrophilic/lipophilic balance (HLB) greater than 10, and the critical micelle concentration (cmc) less than 2000 ppm.

Wetting agent/ surfactant		1.0 % to 3.0 %
Indicator dye	Toluidine blue ^A	0.05 %
Carrier	Distilled water	96.95 % to 98.95 %

^A The chemical name for Toluidine blue is 3-Amino-7-(dimethylamino)-2-methylphenothiazin-5-ium chloride.

5.6.1 Because of the viscosity of many surfactants, 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 surfactant is added to the distilled water by weight and the mixture gently stirred. Vigorous stirring or shaking is not recommended as it may cause foaming that may be difficult to dissipate. Once the surfactant 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 or the surfactant, or both (including options that fall outside the above recommended surface tension, HLB, or CMC ranges) 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 (that is, a “skirt”),