Designation: F1929 – 12

StandardTest Method for Detecting Seal Leaks in Porous Medical Packaging by Dye Penetration[1]

This standard is issued under the fixed designation F1929; 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 This test method defines materials and procedures that will detect and locate a leak equal to or greater than a channel formed by a 50 µm (0.002 in.) wire in package edge seals formed between a transparent material and a porous sheet material. A dye penetrant solution is applied locally to the seal edge to be tested for leaks. After contact with the dye penetrant for a specified time, the package is visually inspected for dye penetration.

1.2 Three dye application methods are covered in this test method: injection, edge dip, and eyedropper.

1.3 These test methods are intended for use on packages with edge seals formed between a transparent material and a porous sheet material. The test methods are limited to porous materials which can retain the dye penetrant solution and prevent it from discoloring the seal area for a minimum of 5 seconds. Uncoated papers are especially susceptible to leakage and must be evaluated carefully for use with each test method.

1.4 These test methods require that the dye penetrant solution have good contrast to the opaque packaging material.

1.5 The values are stated in International System of Units (SI units) and English units. Either is to be regarded as 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]

F17 Terminology Relating to Flexible Barrier Packaging

2.2 *ANSI Standards:*[3]

Z1.4 Sampling Procedures and Tables for Inspection by Attributes

3. Terminology

3.1 *wicking*—the migration of a liquid into the body of a fibrous material. This is distinct from a leak as defined in Terminology F17.

3.2 *dye penetrant*—an aqueous solution of a dye and a surfactant designed to penetrate and indicate a defect location in the time prior to the onset of wicking which could mask its presence.

3.3 *channel*—refer to definition in F17.

4. Significance and Use

4.1 Harmful biological or particulate contaminants may enter the medical package through leaks. These leaks are frequently found at seals between package components of the same or dissimilar materials. Leaks may also result from a pinhole in the packaging material.

4.2 It is the objective of this test method to visually observe the presence of channel defects by the leakage of dye through them.

4.3 This dye penetrant procedure is applicable only to individual leaks in a package seal. The presence of a number of small leaks, as found in porous packaging material, which could be detected by other techniques, will not be indicated.

4.4 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 leaks, components that exhibit any indication of leakage are normally rejected.

4.5 These procedures are suitable to verify and locate leakage sites. They are not quantitative. No indication of leak size can be inferred from these tests. The methods are usually employed as a pass/fail test.

4.6 The dye solution will wick through any porous material over time, but usually not within the maximum time suggested.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packagingand 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.

If wicking does occur, it may be verified by observing the porous side of the subject seal area. The dye will have discolored the surface of the material. Refer to Appendix X1 for details on wicking and guidance on the observance of false positives.

5. Apparatus

5.1 Means of breaching one of the packaging materials such as a small knife. (Method A)

5.2 *Dye Dispenser,* such as an eyedropper or syringe for injection of the dye penetrant solution. (Method A)

5.3 *Dye Solution Container.* (Method B)

5.4 *Scissors* or other cutting instrument. (Method B)

5.5 *Eyedropper* or 1 Mil. Pipette. (Method C)

5.6 *Microscope* or optical loop with magnification of 5× to 20× (optional for all methods).

5.7 Aqueous dye penetrant solution consisting of, by weight:

Wetting agent:	TRITON X-100[4]	0.5 %
Indicator dye:	Toluidine blue	0.05 %
Carrier:	Water	99.45 %

NOTE 1—The solution must remain homogeneous. If precipitate is noted, the solution must be replaced.

5.7.1 If other colored or fluorescent dyes are substituted for toluidine blue, their precision and bias must be experimentally determined.

5.7.2 Because of the viscosity of the TRITON X-100, the preparation of the solution is most easily accomplished by first taring a container with about 10 % of the required amount of water on a scale. The appropriate amount of TRITON X-100 is added to the water by weight and the mixture stirred or shaken. Once the TRITON X-100 is dispersed, the remaining water can then be added, followed by the toluidine blue dye.

6. Safety Precautions

6.1 Injecting dye penetrant into a package with a hypodermic needle and syringe is a common method for performing this test. This practice can result in puncture of the skin with a contaminated needle and is therefore not recommended. Because of this hazard, it is recommended that the dye penetrant is dispensed using a flexible tube attached to a syringe through an opening formed with an appropriate cutting instrument.

7. Test Specimen

7.1 The test specimen shall consist of a complete packaged device, empty packages, or edge seal samples. Blemished, rejected or dummy products may be used if they will not affect test results and are recorded prior to the test.

8. Calibration and Standardization

8.1 Since these procedures are not quantitative, no calibration is required.

[4] TRITON, a registered trademark of Union Carbide, has been found satisfactory for this purpose.

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 the results. See ANSI ASQC Z1.4.

10. Conditioning

10.1 Packaging must be free of condensation or any other source of liquid water. Water already in the seal defects 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 If conditioning is required standard conditioning atmosphere of 23 ± 2°C or 73.4 ± 3.6°F and 50 ± 2 % relative humidity is recommended, for a minimum of 24 hr. prior to testing.

11. Procedure

11.1 *Method A (Injection Method):*

11.1.1 Inject sufficient dye penetrant into the package to cover the longest edge to a depth of approximately 5 mm or 0.25 in. (see 6.1 for safety precautions).

11.1.1.1 When 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 device inside the package. The device 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 Visually examine the seal area through the transparent side of the package. Observe the package seal area for penetration of the dye solution across the seal width. Channels in the seal will be readily detected. Use 5 seconds per side max as a guide for a 4 sided package. Total time should be less than or equal to 20 seconds. With prolonged exposure wicking of dye through the porous packaging will color the entire seal making defect detection difficult. An optical device with 5× to 20× magnification may be used for detailed examination.

11.1.3 Rotate the package as necessary to expose each seal edge to the dye penetrant solution. Inject additional dye as needed to insure complete edge exposure.

11.2 *Method B (Edge Dip Method):*

11.2.1 Select a container whose length is long enough to accommodate the longest sealed edge of the package.

11.2.2 Pour enough dye into the container to cover the entire bottom surface to a minimum depth of approximately 3–6 mm or 0.125–0.25 in.

11.2.2.1 If the package being tested has excessive material beyond the seal, such as a chevron style opening feature, a modification must be made to the package. With a cutting instrument, remove the excessive material along the outside edge of the chevron seal to a distance of approximately 3 mm or 0.125 in. from the seal, taking care not to cut into the seal area. Removal of the excess material will allow the dye solution to come into close proximity to the seal.

11.2.3 Lower one of the edges of the package into the dye solution so that it briefly touches the dye along the entire edge

of the seal. This needs to be a brief dip process, just long enough to completely wet the edge.

11.2.4 Remove the package in its dipped orientation, and verify that the entire seal edge has been exposed to the dye solution. Observe the package seal area, through the transparent side, for penetration of the dye solution across the seal width. Use 5 seconds per side max as a guide for a 4 sided package. Total time should be less than or equal to 20 seconds.

11.2.5 An optical device with 5× to 20× magnification may be used for detailed examination.

11.2.6 Repeat edge dip for the remaining seals.

11.3 *Method C (Eyedropper Method):*

NOTE 2—This method requires the package to have an unsealed area beyond the outer edge of the seal.

11.3.1 Pour dye solution into an open container.

11.3.2 Using a finger or the end of a paper clip, carefully push back the extended edge of the porous material away from the transparent material.

11.3.3 Insert eyedropper or pipette into the dye solution.

11.3.4 With the transparent side of the package facing the operator, lay a bead of the dye solution along the top edge of the package between the porous and transparent material. Ensure entire edge has been wetted with the dye solution.

11.3.5 For small packages slowly rotate the package, while applying solution until the entire package seal is exposed to the solution. Otherwise, apply solution to one side of the package at a time.

11.3.6 Observe the package seal area for penetration of the dye solution across the seal width. Use 5 seconds per side max as a guide for a 4 sided package. Total time should be less than or equal to 20 seconds.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of material being tested, including, but not limited to lot number and source of material, date, time, location and operator of test.

12.1.2 Any conditioning of the materials.

12.1.3 A reference to test method performed: Method A, B, and/or C.

12.1.4 Identification of the dye penetrant solution if different from that specified in section 5.7.

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 or to the interior of the seal via a defined channel shall be taken as an indication of the presence of a leakage site.

12.1.6.2 Evidence of dye penetration through the porous material through general wetting of the surface (wicking) shall not be taken as an indication of the presence of a leakage site.

12.1.6.3 A qualitative description or sketch of the leakage sites.

12.1.6.4 Any deviation from standard.

13. Precision and Bias

13.1 *Injection Method:*

13.1.1 Between June 1997, and March 1998 test packages from four manufacturers were examined using this method by three independent laboratories. Defects were intentionally created in the package seals by placing wires of 50 µm (0.002 in.) diameter in the seal area. When the wires were removed, a channel approximately the size of the wire was created in the seal. For each specimen set, 50 packages were produced, 25 with wire created defects and 25 controls with no artificial defects. The results are shown in Table 1 as (the number of correctly identified defects)/ (the number of test packages).

13.1.2 The results show that when using the dye penetrant on packages with one side consisting of a porous breathable membrane, there is more than 95 % confidence that channels in package seals will be detected if they are equivalent in size in those made with a 50 µm (0.002 in.) wire. In this test series, significant reductions in test performance (probability of detecting a defect <60 %) were observed with pouches fabricated with film on both surfaces and with indicator dyes other than toluidine blue. Previous testing had shown significantly poorer detection with other wetting agents. These test results are therefore specific for this dye and wetting agent formulation.

13.1.3 The above P&B statement and Table 1 were generated using Method A only.

13.1.4 *Bias*—Pass/fail tests have no bias.

13.2 *Edge Dip and Eyedropper Methods:*

13.2.1 Edge dip and eyedropper Interlaboratory studies of ASTM F1929, Standard Test Method for Detecting Seal Leaks in Porous Medical Packaging by Dye Penetration were conducted in 2012. Of the twelve laboratories that participated, seven tested the edge dip method, and five tested the eyedropper method. Defects were intentionally created by placing wires of 50 µm (0.002 in.) diameter in the seal area. The wires were removed and a channel approximately the size of the wire was created in the seal. Each participant analyzed 50 randomly coded samples (25 produced with channels and 25 without

TABLE 1 Results on Testing Seals with Channels Generated Using 50 µm (0.002 in.) Wires

Test Site	1	2	3	
Sample 1: Breathable pouch; coated 44# paper				
With defect	25/25	24/25	22/24	
No defect	24/24	24/24	25/25	
Sample 2: Tray with breathable lid; dot coated TYVEK[A]				
With defect	25/25	25/25	24/25	
No defect	25/25	25/25	25/25	
Sample 3: Breathable pouch; coated TYVEK				
With defect	25/25	25/25	24/24	
No defect	23/25	25/25	25/25	
Sample 4: Breathable pouch; dot coated TYVEK				
With defect	24/25	25/25	25/25	25/25[B]
No defect	25/25	25/25	25/25	25/25
Summary				
			Defective	No Defect
Number correctly identified			318	321
Total tested			323	323
Percent correctly identified			98 %	99 %

[A] TYVEK, a registered trademark of DuPont, has been found satisfactory for this purpose.
[B] Tested at manufacturing site.