Designation: F1929 – 15

Standard Test 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.

Current edition approved Oct. 1, 2015. Published December 2015. Originally approved in 1998. Last previous edition approved in 2012 as F1929 – 12. DOI: 10.1520/F1929-15.

[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 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.

channels) for each of five materials. Every analyst reported results to indicate the presence or absence of a channel. The results were tabulated as the number identified correctly, false positives, and false negatives; the details are given in RR:F02-1032.[5]

13.2.2 When combining the edge dip data population of all labs, the results show that this method provides the correct response of detecting channels created by a 50 µm wire in seals 95 % of the time. The 95 % confidence interval is 93.8 % to 96.0 %. The results of correctly identified, false positives and false negatives are shown in Tables 2-4.

13.2.2.1 When comparing the edge dip labs through ANOVA, labs 6520 and 6521 were significantly different than the other five. This would suggest that there is potentially some other cause of their greater error rate. If they were excluded, the rate improves to 98 % (96.8 % to 98.5 % at a 95 % confidence level).

13.2.2.2 Comments noted by Lab 6521 described observing some samples having a distinct pathway highlighted by the dye, but only partially traversing the seal width. These samples were not recorded as channels. The reason was that the definition of a channel requires a complete passage of the dye through the seal. All of the partial channels for this lab, when compared to the defect failure key, were confirmed to be channels. For lab 6521, these samples contributed to a significantly increased false negatives response.

13.2.3 When combining the eyedropper data population of all labs, the results show that this method provides the correct response of detecting channels created by a 50 µm wire channels in seals 99 % of the time. The 95 % confidence interval is 97.7 % to 99.1 %. The results of correctly identified, false positives and false negatives are shown in Tables 5-7.

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

13.2.5 The materials tested were identified as:

(1) Coated Tyvek, Hot Melt Adh., /Rigid Packaging Substrate.

(2) Uncoated Tyvek, No Adh., /Flexible Packaging Substrate.

(3) Coated Tyvek, Hot Melt Adh., /Flexible Packaging Substrate.

(4) Coated Tyvek, Water-Based Adh., /Flexible Packaging Substrate.

(5) Coated Paper, Water-Based Adh., /Flexible Packaging Substrate.

14. Keywords

14.1 dye penetrant; edge dip; eyedropper; flexible packaging; porous packaging; seal leaks

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1032. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Edge Dip – correctly identified

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6512	100%	100%	100%	100%	100%	100.0%	0.00
6518	92%	100%	94%	100%	100%	97.2%	3.90
6520	84%	94%	78%	94%	86%	87.2%	6.87
6521	94%	86%	78%	94%	92%	88.8%	6.87
6522	96%	100%	90%	94%	98%	95.6%	3.85
8490	98%	100%	96%	98%	98%	98.0%	1.41
8491	98%	98%	100%	96%	98%	98.0%	1.41
Average	94.6%	96.9%	90.9%	96.6%	96.0%		
Std Dev	5.38	5.27	9.44	2.76	5.16		

TABLE 3 Edge Dip – false positive (channel noted where none existed)

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6512	0%	0%	0%	0%	0%	0.0%	0.00
6518	2%	0%	0%	0%	0%	0.4%	0.89
6520	4%	0%	0%	0%	2%	1.2%	1.79
6521	0%	0%	0%	0%	0%	0.0%	0.00
6522	2%	0%	0%	2%	2%	1.2%	1.10
8490	0%	0%	0%	0%	0%	0.0%	0.00
8491	0%	0%	0%	0%	0%	0.0%	0.00
Average	1.1%	0.0%	0.0%	0.3%	0.6%		
Std Dev	1.57	0.00	0.00	0.76	0.98		

TABLE 4 Edge Dip – false negative (no channel identified where one existed)

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6512	0%	0%	0%	0%	0%	0.0%	0.00
6518	6%	0%	6%	0%	0%	2.4%	3.29
6520	12%	6%	22%	6%	12%	11.6%	6.54
6521	6%	14%	22%	6%	8%	11.2%	6.87
6522	2%	0%	10%	4%	0%	3.2%	4.15
8490	2%	0%	4%	2%	2%	2.0%	1.41
8491	2%	2%	0%	4%	2%	2.0%	1.41
Average	4.3%	3.1%	9.1%	3.1%	3.4%		
Std Dev	4.07	5.27	9.44	2.54	4.72		

TABLE 5 Eyedropper Method – correctly identified

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6510	100%	100%	96%	100%	100%	99.2%	1.79
6511	98%	100%	98%	98%	100%	98.8%	1.10
6513	100%	100%	98%	96%	98%	98.4%	1.67
6514	100%	100%	94%	94%	100%	97.6%	3.29
6516	98%	100%	96%	100%	100%	98.8%	1.79
Average	99.2%	100.0%	96.4%	97.6%	99.6%		
Std Dev	1.10	0.00	1.67	2.61	0.89		

TABLE 6 Eyedropper Method – false positive (channel noted where none existed)

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6510	0%	0%	2%	0%	0%	0.4%	0.89
6511	0%	0%	0%	0%	0%	0.0%	0.00
6513	0%	0%	0%	0%	0%	0.0%	0.00
6514	0%	0%	2%	2%	0%	0.8%	1.10
6516	2%	0%	0%	0%	0%	0.4%	0.89
Average	0.4%	0.0%	0.8%	0.4%	0.0%		
Std Dev	0.89	0.00	1.10	0.89	0.00		

TABLE 7 Eyedropper Method – false negative (no channel identified where one existed)

Laboratory	Samples					Average	Std Dev %
	Material 1	Material 2	Material 3	Material 4	Material 5		
6510	0%	0%	2%	0%	0%	0.4%	0.89
6511	2%	0%	2%	2%	0%	1.2%	1.10
6513	0%	0%	2%	4%	2%	1.6%	1.67
6514	0%	0%	4%	4%	0%	1.6%	2.19
6516	0%	0%	4%	0%	0%	0.8%	1.79
Average	0.4%	0.0%	2.8%	2.0%	0.4%		
Std Dev	0.89	0.00	1.10	2.00	0.89		

APPENDIX

(Nonmandatory Information)

X1. FALSE POSITIVE GUIDANCE

X1.1 *Wicking*—The dye solution used for this testing is very aggressive. It will wick along the fibers of the Tyvek® and other porous materials quickly. This will appear to be a partial penetration of the seal area, and can be misinterpreted as a test failure. Dissimilarly, a channel defect in the seal provides a path across the entire seal width and will be evident almost immediately after exposure to the dye solution. This can be readily distinguished from the wicking phenomenon, as capillary action in channel is much faster than wicking. The correct exposure time is important in distinguishing the difference between wicking and a channel. 5 second is all the time required, since channel penetration is typically noticed with in the first few seconds of exposure. If dye solution exposure is for 20 seconds, wicking can be severe. 20 seconds is the intended exposure time for the whole package, i.e. 5 seconds × 4 sides = 20 seconds.

X1.2 *Oxidative Sterilization*—Oxidative sterilization processes can change the hydrostatic head of some porous materials. This change to the surface tension can cause false positive results due to excessive wicking. Before using liquid based integrity testing, it is important to evaluate the sterilization effects on the hydrostatic head property of the materials.

X1.3 *Bending/Folding*[6]—A false-positive can occur when a flexible porous material is bent, folded, or creased. The folding causes internal sheet separation of the porous web. This can happen when a pouch is folded to fit into a shelf container or is folded or bent during distribution stress testing. The folding of porous barrier materials is not recommended but is often ignored or difficult to avoid. Sheet separation has been observed in all types of porous sheet materials. Porous sheets can separate internally because the exterior surfaces are less flexible than the interior. The bending forces can result in the yielding of the interior fibers that hold the sheet together. The tighter the bend the greater forces become until the load becomes excessive and the fiber structure holding the sheet together will separate and compress on one side of the bend, expand on the other side creating a gap or channel between internal fibers at the bend. When the sheet is unbent or flattened out again, there will still be a less dense area or gap formed in the interior of the sheet. These areas in the porous sheet are separations within the softer inner layer between the more rigid outer surfaces. The original mass of the fibers are still there, only the bulk density has decreased. During dye testing, the dye will wick through the surface layer and penetrate to the separation site. At that point, the dye finds this path of least resistance and quickly migrates to the edge of the sheet creating a channel like appearance. The study published in the referenced article can aid the reader in identifying this false-positive phenomenon.

[6] Curtis L. Larsen "Porous Sterile Barrier Integrity Testing: Failure Anomalies" *Medical Device & Diagnostic Industry*; January 2006.