Designation: ~~F1929 – 98 (Reapproved 2004)~~ F1929 – 12

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 ~~a procedure~~ 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 ~~film~~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 ~~This~~These test ~~method is~~methods are intended for use on packages with edge seals formed between a transparent ~~film~~material and a porous sheet material. ~~This~~The test ~~method is~~methods are limited to porous materials which can retain the dye penetrant solution and prevent it from discoloring the ~~entire~~ seal area for a minimum of ~~20 s.~~5 seconds. Uncoated papers are especially susceptible to leakage and must be evaluated carefully ~~fro~~for use with ~~this~~each test method.

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

1.5 The values ~~stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.~~ 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]

~~F1327~~F17 Terminology Relating to ~~Barrier Materials for Medical~~ Flexible Barrier Packaging ~~(Withdrawn 2007)~~

2.2 *ANSI Standards:*[3]

Z1.4 Sampling Procedures and Tables for Inspection by Attributes

3. Terminology

3.1 *wicking*—~~The~~the migration of a liquid into the body of a fibrous material. This is distinct from a leak as defined in Terminology ~~F1327~~F17.

3.2 *dye penetrant*—~~An~~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*—~~A small continuous open passage across the width of a package seal through which microorganisms could pass. It is the objective of this test method to visually observe~~refer to definition in F17~~the presence of these defects by the leakage of dye through them.~~.

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

Current edition approved ~~June 1, 2004~~Nov. 1, 2012. Published ~~June 2004~~January 2013. Originally approved in 1998. Last previous edition approved in ~~1998~~2004 as ~~F1929 – 98.~~F1929 – 98(2004). DOI: ~~10.1520/F1929-98R04.~~10.1520/F1929-12.

[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.

4. Significance and Use

4.1 Harmful biological or particulate contaminants may enter the ~~device~~ 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 ~~leakage,~~leaks, components that exhibit any indication of leakage are normally rejected.

4.5 ~~Since leaks may change in size with different ambient conditions, comparisons between test stations are not conclusive. Therefore this method is~~ 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 ~~go, no-go~~ pass/fail test.

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

~~4.6 When puncturing the packaging to allow injection of the dye penetrant solution, care should be taken not to puncture 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.~~

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×.~~20× (optional for all methods).

~~5.4 Fresh aqueous dye penetrant solution consisting of, by weight:~~

~~Wetting agent:~~	~~TRITON X-100[5]~~	~~0.5 %~~
~~Indicator dye:~~	~~Toluidine blue~~	~~0.05 %~~

~~5.5 Other colored or fluorescent dyes may be substituted for toluidine blue but their precision and bias must be experimentally determined.~~

5.7 Aqueous dye penetrant solution consisting of, by weight:

~~Because~~

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

~~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.~~

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

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

hazard, it is recommended that the dye penetrant ~~be~~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 if the test will be used as a quality control method. Blemished or rejected~~ device, empty packages, or edge seal samples. Blemished, rejected or dummy products may be used if ~~the defect~~ they will not affect test results and ~~is~~are recorded prior to the test.

~~7.2 Dummy test items, empty packages, or edge seal samples may be used for process control, product acceptance, or material development testing.~~

8. Calibration and Standardization

8.1 ~~These procedures are suitable for use on selected parts during receiving inspection or to verify and locate leakage sites for production control. They are not quantitative. No indication of leak size can be inferred from the test.~~Since these procedures are not quantitative, no calibration is required.

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 ~~Test specimens shall be conditioned prior to testing. When no specific conditioning requirements are given, and packaging materials are moisture sensitive, a~~ If conditioning is required standard conditioning atmosphere of 23 ± 2°C ~~(73.4~~ or 73.4 ± ~~3.6°F)~~3.6°F and 50 ± 2 % relative humidity is recommended, for a minimum of 24 ~~h~~hr. prior to testing.

11. Procedure

~~10.1 Cleaning of packaging prior to dye penetrant application is unnecessary.~~

11.1 ~~Inject sufficient dye penetrant into the package to cover the longest edge to a depth of approximately 5 mm (0.25 in.). Allow the dye penetrant solution to remain in contact with the seal edge for a minimum of 5 s and a maximum of 20. Channels will be detected within this time period but beyond 20 s, wicking of dye through the porous packaging will color the entire seal.~~*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.

~~10.3 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 ~~Visually examine the seal area through the transparent side of the package. Channels in the seal will be readily apparent without magnification, as the dye will rapidly wick into the area adjacent to the channel, making a much larger stain than the actual channel size. An optical device with 7× to 20× magnification may be used for detailed examination.~~ *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.

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.

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 ~~this test method,~~test method performed: Method A, B, and/or C.

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

12.1.5 Method of ~~inspection, and~~visual inspection: aided or unaided.

12.1.6 Results: