Designation: F 1929 – 98 (Reapproved 2004)

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

This standard is issued under the fixed designation F 1929; 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 that will detect and locate a leak equal or greater than a channel formed by a 50 µm (0.002 in.) wire in package edge seals formed between a transparent film 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 This test method is intended for use on packages with edge seals formed between a transparent film and a porous sheet material. This test method is 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. Uncoated papers are especially susceptible to leakage and must be evaluated carefully fro use with this test method.

1.3 This test method requires that the dye penetrant have good contrast to the opaque packaging material.

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

1.5 *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]

F 1327 Terminology Relating to Barrier Materials for Medical 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 F 1327.

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*—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 the presence of these defects by the leakage of dye through them.

4. Significance and Use

4.1 Harmful biological or particulate contaminants may enter the device 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 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.

4.3 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 are normally rejected.

4.4 Since leaks may change in size with different ambient conditions, comparisons between test stations are not conclusive. Therefore this method is usually employed as a go, no-go test.

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[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.60 on Medical Device Packaging.

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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.

4.5 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.

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.

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

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

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

Wetting agent:	TRITON X-100[4]	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.

5.6 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 be 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 products may be used if the defect will not affect test results and is 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.

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

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.

9. Conditioning

9.1 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.

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

10. Procedure

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

10.2 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.

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.

10.4 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.

11. Report

11.1 Report the following information:

11.1.1 A reference to this test method,

11.1.2 Identification of the dye penetrant,

11.1.3 Method of inspection, and

11.1.4 Results:

11.1.4.1 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 the indication of the presence of a leakage site.

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

11.1.4.3 A qualitative description or sketch of the leakage sites.

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. Precision and Bias

12.1 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).

12.2 The results show that when using the dye penetrant as specified in this test methodology on packages with one side consisting of a porous breathable membrane there is better than 95 % confidence that channels in package seals, equivalent in size in those made with a 50 µm (0.002 in.) wire will be detected. 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. Keywords

13.1 Dye penetrant; flexible packaging; porous packaging; seal leaks