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.

Designation: F2096 – 11

Standard Test Method for Detecting Gross Leaks in Packaging by Internal Pressurization (Bubble Test)[1]

This standard is issued under the fixed designation F2096; 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 covers the detection of gross leaks in packaging. Method sensitivity is down to 250 µm (0.010 in.) with an 81 % probability (see Section 11). This test method may be used for tray and pouch packages.

1.2 The sensitivity of this test method has not been evaluated for use with porous materials other than spunbonded polyolefin or with nonporous packaging.

1.3 This test method is destructive in that it requires entry into the package to supply an internal air pressure

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]

F17 Terminology Relating to Flexible Barrier Packaging

3. Terminology

3.1 *Definitions*—General terms relating to barrier materials for medical packaging are found in Terminology F17.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *breathing point pressure, n*—pressure at which permeation of air through the porous material begins.[3]

4. Summary of Test Method

4.1 The package is inflated underwater to a predetermined pressure. The package is then observed for a steady stream of air bubbles indicating a failure area.

4.2 The sensitivity of this test method is dependent on the differential pressure and method of pressurization. Establishment of a test pressure for each package material/size is critical for obtaining repeatable results (see Annex A1 for the procedure on establishing test pressure). Inadequate pressurization of the package can significantly reduce the sensitivity of this test method. Higher differential pressures will increase the test sensitivity. However, excessive pressurization of the package may rupture seals or cause misinterpretation of bubble patterns emanating from porous packaging. This may result in an erroneous conclusion regarding the presence or absence of package defects. While not required, use of a bleed-off control valve in line with the pressure monitoring device, will aid in stabilizing the test pressure, and help eliminate excessive pressurization of the package (see Fig. 1).

4.3 Two different test methods are presented for the testing of porous and nonporous packaging. The key difference between the test methods (as described in Annex A1) is in allowing time for the water to saturate the porous material.

5. Significance and Use

5.1 The internal pressurization test method provides a practical way to examine packages for gross leaks.

5.2 This test method is extremely useful in a test laboratory environment where no common package material/size exists.

5.3 This test method may apply to large or long packages that do not fit into any other package integrity test method apparatus.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and 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] All porous packaging by definition will permit the passage of air. At a given internal pressure it will therefore exhibit an emanating stream of air bubbles dependent on the pore size. A stream of bubbles identified at a lower internal pressure than the breathing pressure point may indicate a defect in the packaging.

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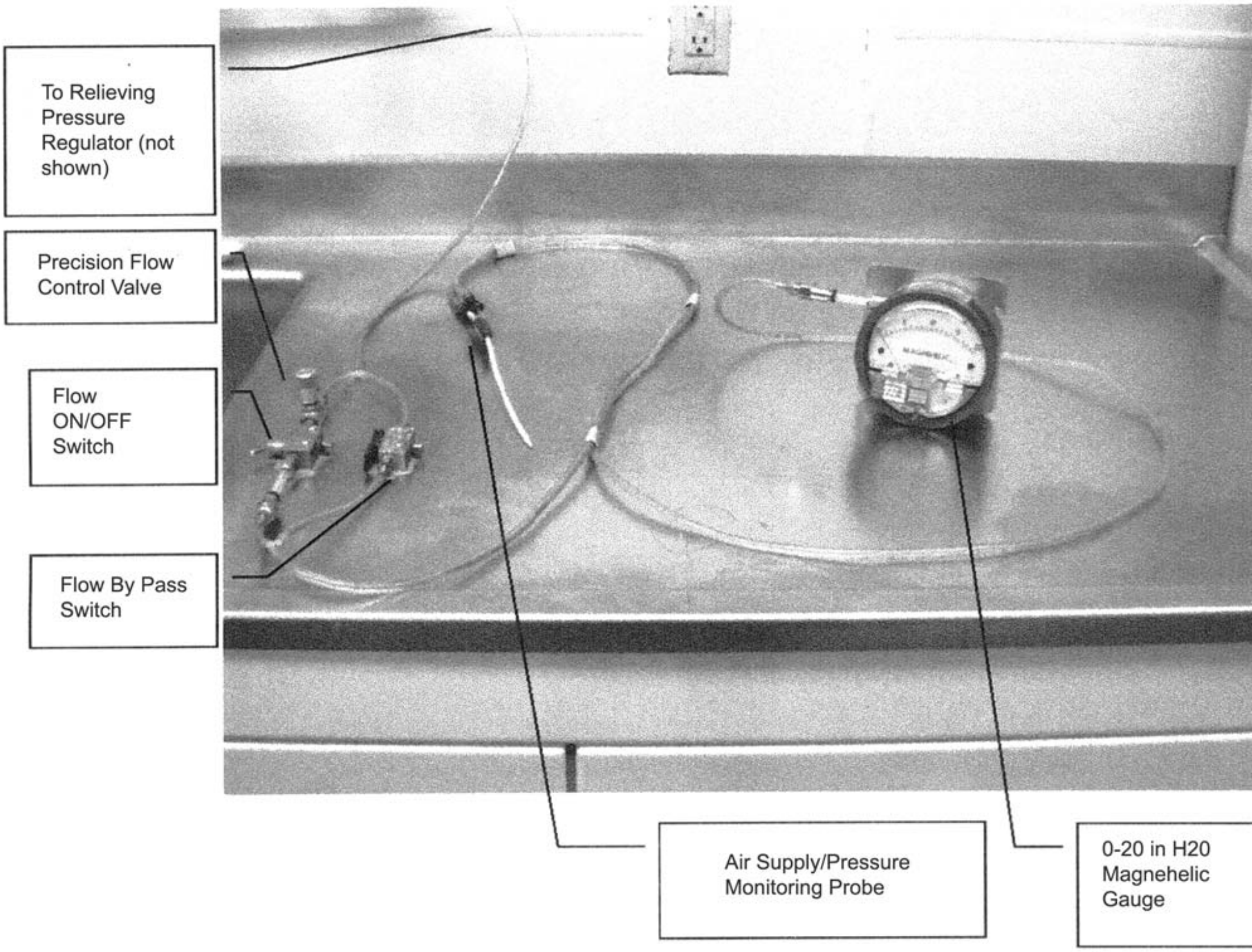

FIG. 1 Sample Test Apparatus

5.4 This test method may be used as a means to evaluate package integrity. Package integrity is crucial to consumer safety since heat sealed packages are designed to provide a contamination free or sterile environment, or both, to the product.

5.5 This test method may be used to detect substrate holes and channels.

6. Apparatus

6.1 *Pressure Delivery System,* with pressure monitoring gage, and bleed-off control valve, capable of delivering air at a pressure of 0-50 mbar (0-20 in. H_2O).

6.2 *Device for Puncturing Package,* device to allow insertion of air source and pressure monitoring device.

6.3 *Water Container,* adequate to cover the test specimen with approximately 25.4 mm (1 in.) of water.

NOTE 1—It may be beneficial for observation of the test specimen and for interpretation of results to perform the testing in a water container that has at least one transparent side.

7. Sampling

7.1 The number of test specimens shall be chosen to permit an adequate determination of representative performance.

8. Conditioning

8.1 No special conditioning of the specimen is required.

9. Procedure

NOTE 2—The establishment of a test pressure in accordance with Annex A1 must be performed prior to initiating the test procedure. It is recommended that a sample test set-up be provided.

9.1 *Test Method A—Procedure for Nonporous Packaging:*

9.1.1 Create a hole in the package using a puncturing device for inserting the air source and pressure monitor into the control sample. Create the hole wherever it is most efficient to observe defects without obscuring any pre-existing defects or creating defects in the inner package during the puncturing process. The hole size should allow insertion of the air source and pressure monitor with minimal air leakage. Use tape or a rubber disk as a septum over the puncture site to seal the insertion site if necessary.

9.1.2 Insert the air source and pressure monitor into the test specimen. Submerge the package approximately 1 in. under water. Start airflow into the package.

NOTE 3—It may be helpful to use a fixture to keep the entire package submerged at the proper depth.

9.1.3 Adjust the airflow and bleed-off valve as necessary to slowly inflate the package to a value equal to or greater than the

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minimum test pressure as established in accordance with Annex A1. Adjust the bleed-off valve and pressure regulator as necessary to maintain constant pressure.

9.1.4 Thoroughly inspect one side of the package facing upwards for a constant stream of bubbles indicating a specific area of failure (seal channels, pinholes, cracks, tears, and so forth). Then repeat the process by rotating the package 180º so the opposite side of the package is facing upwards. Inspection time will vary depending on package size.

9.1.5 Remove the package from water and mark any observed area(s) of failure.

9.2 *Test Method B—Procedure for Porous Packaging:*

9.2.1 Apply blocking agent to samples if required in accordance with A1.1.2.4.

9.2.2 Create a hole in the package using a puncturing device for inserting the air source and pressure monitor into the control sample. Create the hole wherever it is most efficient to observe defects without obscuring any pre-existing defects or creating defects in the inner package during the puncturing process. The hole size should allow insertion of the air source and pressure monitor with minimal air leakage. Use tape or a rubber disk as a septum over the puncture site to seal the insertion site if necessary.

9.2.3 Insert the air source and pressure monitor into the package. Submerge the package approximately 25.4 mm (1 in.) under water with the porous part of the package in the up position (if one side is porous) and hold for a minimum of 5 s. Start the airflow into the package.

NOTE 4—It may be helpful to use a fixture to keep the entire package submerged at the proper depth.

9.2.4 Adjust the airflow and bleed-off valve as necessary to slowly inflate the package to a value equal to or greater than the minimum test pressure as established in accordance with Annex A1. Adjust the bleed-off valve and pressure regulator as necessary to maintain constant pressure.

9.2.5 Thoroughly inspect the porous side of the package facing upwards for a constant stream of bubbles indicating a specific area of failure (seal channels, pinholes, cracks, tears, and so forth). Then repeat this process by rotating the package 180º so the opposite side of the package is facing upwards. Inspection time will vary depending on package size.

NOTE 5—For packages that may be susceptible to premature deterioration from extended exposure to water, rotation of the package is optional. However, thorough inspection of both surfaces of the package is required.

9.2.6 Remove the package from the water and mark any observed area(s) of failure.

10. Report

10.1 Report the following information:

10.1.1 Date and operator's name or initials.

10.1.2 Package type and any applicable traceable identification numbers.

10.1.3 Established defect size used to establish the test sensitivity, test pressure, and use of any blocking agent.

10.1.4 Number of test packages, number of packages that passed, number of packages exhibiting leaks, and location of each leak

10.1.5 Record the instrument used to create the opening of the package.

11. Precision and Bias

11.1 A round-robin study was conducted in 2000, which included 5 laboratories, two package types, and two defect types. The defects consisted of a channel through the seal area on the pouch samples and a puncture through the porous material, on both the tray and pouch samples. All defect sample groups were created with a 125-µm (0.005 in.) and 250-µm (0.010 in.) wire. The first package type consisted of a 4 by 6-in. heat-sealed pouch, made from a combination clear film and uncoated Tyvek.[4] The second package type consisted of a 3 by 5-in. thermoformed polycarbonate tray, heat-sealed with an adhesive zone coated Tyvek. The negative controls consisted of the same packages produced with no defects. For each specimen set, 15 samples were produced, 10 with defects, and 5 controls with no defect. The results are presented in Table 1 and the corresponding graph in Fig. 2.

11.2 The analysis of the testing process is by means of logistic regression. The computations were performed in SAS, version 8.1. The *y*-axis is the probability of rejecting a part, and the *x*-axis is the nominal defect size. The results of this analysis indicate that there is an 81 % probability of rejecting a package with a defect size of 250 µm. There is also an 11 % probability of rejecting a package with no defects.

11.3 Pass/fail tests have no bias.

[4] Tyvek is a registered trademark of Dupont and has been found satisfactory for this purpose.

TABLE 1 Percent Correct by Laboratory and Defect Type

Laboratory	Defect Type								Percent Correct by Laboratory
	Pouch					Tray			
	No Defects	125-µm Channel	250-µm Channel	125-µm Puncture	250-µm Puncture	No Defects	125-µm Puncture	250-µm Puncture	
1	100	100	90	60	80	100	40	90	82.50
2	100	70	90	50	60	90	10	100	71.25
3	80	20	80	60	80	90	60	80	68.75
4	100	70	90	0	0	100	60	100	65.00
5	80	20	100	0	30	100	20	90	55.00
Percent Correct by Defect	92	56	90	34	50	96	38	92	

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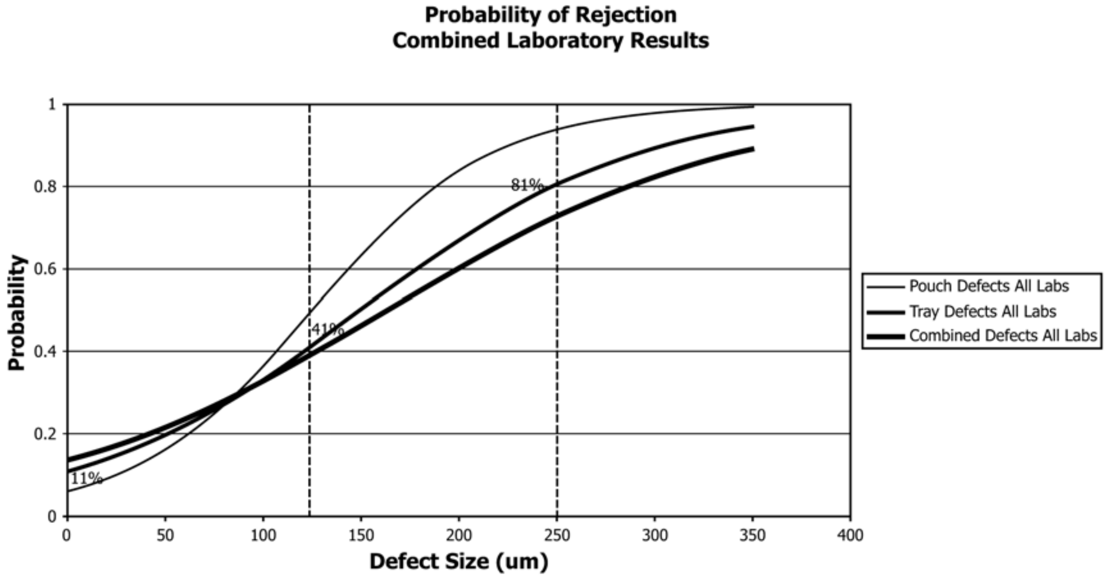

FIG. 2 Probability of Rejection-Combined Laboratory Results

12. Keywords

12.1 bubble test; internal pressurization; package integrity

ANNEX

(Mandatory Information)

A1. ESTABLISHMENT OF TEST PRESSURE

A1.1 This test method uses a control sample with a known defect for establishing the test pressure for the package material/size. A known defect is created in the package surface. The package is submerged in water and inflated to a pressure where air bubbles are observed emanating from the defect. This is considered the minimum test pressure for the given package. Inadequate pressurization of the package can significantly reduce the sensitivity of the test method. Higher differential pressures will increase the test sensitivity. However, excessive pressurization of the package may rupture seals or cause misinterpretation of bubble patterns emanating from porous packaging. This may result in an erroneous conclusion regarding the presence or absence of package defects.

During feasibility studies, it was noted that the pressure at which the permeation of air through the porous material began (breathing point pressure), was significantly less when a positive airflow was introduced into the package before submersion. A study was performed to determine the effects of water permeation on the breathing point pressure of the Tyvek. A positive airflow was introduced into a test sample before submersion. When the package was submerged in water, at a depth of 25.4 mm (1 in.), the breathing point pressure was recorded at approximately 101.6 to 127.0 mm (4 to 5 in.) H_2O. A test sample was then submerged in water, at a depth of 25.4 mm (1 in.), for a minimum of 5 s, prior to the airflow being introduced into the package. The breathing point pressure increased to approximately 228.6 mm (9 in.) H_2O. The test was then repeated with a known defect produced in the package. The pressure at which the defect was observed was 127 mm (5 in.) H_2O. Introduction of airflow prior to submersion, or after submersion, did not affect the pressure at which the defect was observed. However, due to the permeation of air through the Tyvek, the defect could not be consistently detected when the airflow was started prior to submersion.

A1.1.1 *Establishment of Test Pressure for Nonporous Packaging*

A1.1.1.1 A1.1.1.1 Create a known defect in the control sample to the desired sensitivity for the test method (defect not to exceed 250 µm). Circle the location of the defect with a soft tip permanent pen. Record the known defect size for reporting purposes.

A1.1.1.2 Create a hole in the package using a puncturing device for inserting the air source and pressure monitor into control sample. Create the hole wherever it is most efficient to observe defects without obscuring any pre-exsisting defects or creating defects in the inner package during the puncturing process. The hole size should allow insertion of the air source and pressure monitor with minimal air leakage. Use tape or a rubber disk as a septum over the puncture site to seal the

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insertion site if necessary. The location of the hole should not interfere with the defect created in A1.1.1.1.

A1.1.1.3 Insert the air source and pressure monitor into the control sample. Submerge the control sample under water approximately 25.4 mm (1 in.). Start the airflow into the control sample.

A1.1.1.4 Adjust the airflow and bleed-off valve as necessary, to gradually inflate the control sample to the point where the defect manifests itself. Record the pressure reading. This pressure will be the minimum test pressure. Higher differential pressures will increase the test sensitivity. However, excessive pressurization of the package may cause package seals to creep open or rupture.

A1.1.2 *Establishment of Test Pressure for Porous Material Packaging*

A1.1.2.1 Create a known defect in the control sample to the desired sensitivity for the test method (defect not to exceed 250 µm). Circle the location of the defect with a soft tip permanent pen. Record the defect size for reporting purposes.

A1.1.2.2 Create a hole in the package using a puncturing device for inserting the air source and pressure monitor into the control sample. Create the hole wherever it is most efficient to observe defects without obscuring any pre-existing defects or creating defects in the inner package during the puncturing process. The hole size should allow insertion of the air source and pressure monitor with minimal air leakage. Use tape or a rubber disk as a septum over the puncture site to seal the insertion site, if necessary. The location of the hole should not interfere with the defect created in A1.1.2.1.

A1.1.2.3 Insert the air source and pressure monitor into the control sample Submerge the test specimen under water approximately 25.4 mm (1 in.) and hold for a minimum of 5 s. Start the airflow into the control sample.

A1.1.2.4 Adjust the airflow and bleed-off valve as necessary, to gradually inflate the control sample to the point where the defect manifests itself.

NOTE A1.1—If porous material begins to breath prior to observing the defect, apply a blocking agent[5] to the porous material and repeat A1.1.2.3 and A1.1.2.4. Otherwise, proceed to A1.1.2.5. The intent of the blocking agent is to reduce the porosity of the porous material, therefore, increasing the internal test pressure. This may aid the user in detecting the known defect. The type and quantity and method by which the blocking agent is applied (for example, hand-applied) may have a profound effect on both the physical and chemical nature of the porous packaging material and may therefore give a wide range of breathing point pressures. It is recommended to use the minimal quantity of blocking agent necessary to detect the known defect.

A1.1.2.5 Record the pressure reading. This pressure will be the minimum test pressure. Higher differential pressures will increase the test sensitivity. However, excessive pressurization of the package may rupture seals or cause misinterpretation of bubble patterns emanating from porous packaging.

[5] Steris Alcare Alcohol Foam and Steris Alcare Foamed Alcohol Hand Cleaner has proven effective as a masking agent in raising the breathing pressure point of porous material and has been found satisfactory for this purpose.

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