Designation: F2228 – 13

Standard Test Method for Non-Destructive Detection of Leaks in Packaging Which Incorporates Porous Barrier Material by CO_2 Tracer Gas Method[1]

This standard is issued under the fixed designation F2228; 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 non-destructive test method detects leaks in non-porous rigid thermoformed trays, as well as the seal between the porous lid and the tray. The test method detects channel leaks in packages as small as 100 µm (0.004 in.) diameter in the seal as well as 50 µm (0.002 in.) diameter pinholes, or equivalently sized cracks in the tray, subject to trace gas concentration in the package, package design and manufacturing tolerances.

NOTE 1—This test method does not claim to challenge the porous (breathable) lidding material. Any defects that may exist in the porous portion of the package will not be detected by this test method.

1.2 The values stated in SI units are to be regarded as standard units. Values in parentheses are for information only.

1.3 *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]

D996 Terminology of Packaging and Distribution Environments

F17 Terminology Relating to Flexible Barrier Packaging

F1327 Terminology Relating to Barrier Materials for Medical Packaging (Withdrawn 2007)[3]

3. Terminology

3.1 *General Term Definitions*—For definitions used in this standard, see Terminologies D996, F17, and F1327.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *basal flow*—transverse transport of trace gas across the seal due to gas flow within the plane of the porous barrier material as well as flow between the porous barrier and the temporary gasketing. This is an expected property of the porous barrier material and does not represent a leak. Experimentally, this flow may be thought of as noise, which will always be present, to some degree, during testing and must be accounted for.

3.2.2 *trace gas*—a compound selected solely for use to identify leakage flow.

4. Summary of Test Method

4.1 This test method utilizes CO_2 sensing techniques in the detection of a CO_2 trace gas to quantify leaks in medical packaging, which incorporates porous barrier material. This test method provides a qualitative (accept/reject) inspection method to evaluate packages for pinhole, crack and channel leaks. Further information on the "Leak Test Theory" may be found in Annex A1.

5. Significance and Use

5.1 Harmful biological or particulate contaminants may enter the package through incomplete seals or imperfections such as pinholes or cracks in the trays.

5.2 After initial instrument set-up and calibration, the operations of individual tests and test results do not need operator interpretation. The non-destructive nature of the test may be important when testing high value added products.

5.3 Leak test results that exceed the permissible threshold setting are indicated by audible or visual signal responses, or both, or by other means.

5.4 This non-destructive test method may be performed in either laboratory or production environments. This testing may be undertaken on either a 100 % or a statistical sampling basis. This test method, in single instrument use and current

[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] The last approved version of this historical standard is referenced on www.astm.org.

implementation, may not be fast enough to work on a production packaging line, but is well suited for statistical testing as well as package developmental design work.

6. Apparatus

6.1 *Non-destructive Trace Gas Leak Detection Apparatus*—The apparatus' test fixture consists of three major elements and is shown in Fig. 1.

6.2 *Sealing Membrane*—The purpose of the sealing membrane is to seal off the tracer gas transmission, normal to the porous lid surface. However, the membrane does not completely control the transmission of tracer gas basal flow in the transverse direction.

6.3 *Control Packages*—Packages with calibrated capillary channel leaks as well as packages with calibrated pinholes in the tray constructed for instrument calibration as well as for test procedure verification.

6.4 *Test Fixture*—Apparatus, which must be designed to ensure detection of a calibrated leak.

7. Preparation of Apparatus

7.1 The test apparatus is to be started, warmed-up, and made ready according to the manufacturer's specifications. The instrument must be operated in an environment as described in the instrument's user manual.

8. Reagents and Materials

8.1 *CO_2 Trace Gas Cylinder and Regulator* —A cylinder of "Commercial" or "Bone Dry" grade carbon dioxide with a minimum of 206.84 kPa (30 psi) pressure is required for calibration and testing.

8.2 *Sealing Membrane*—The temporary sealing membrane must exhibit the correct pliability and tackiness in order to form a gas-tight bond with the porous lidding materials during the testing process, and must release at the end of the test without damaging the porous lid or the edge seal.

8.3 *Sealing Membrane-induced Damage*— During the process of membrane selection for a specific package design and configuration, inspect the packages for the following indications of membrane-induced damage after the membrane is removed from the package:

8.3.1 Sticky residue remaining on the porous barrier material at the end of the test cycle.

8.3.2 Fibers from the porous barrier material remaining on the sealing membrane at the end of the test cycle.

8.3.3 Visible changes to the texture or structure of the porous lidding material at the end of the test cycle, under microscope or other magnified examination.

8.3.4 Damage to the printed information on the porous barrier. The adhesive of the sealing membrane may lift off the ink from the barrier.

8.3.5 Failure of the package to release from the sealing membrane at the end of the test cycle.

8.3.6 Damage to the seal incurred on removal of the membrane from the package.

9. Hazards

9.1 As the test fixture is closed, it may present pinch-point hazards.

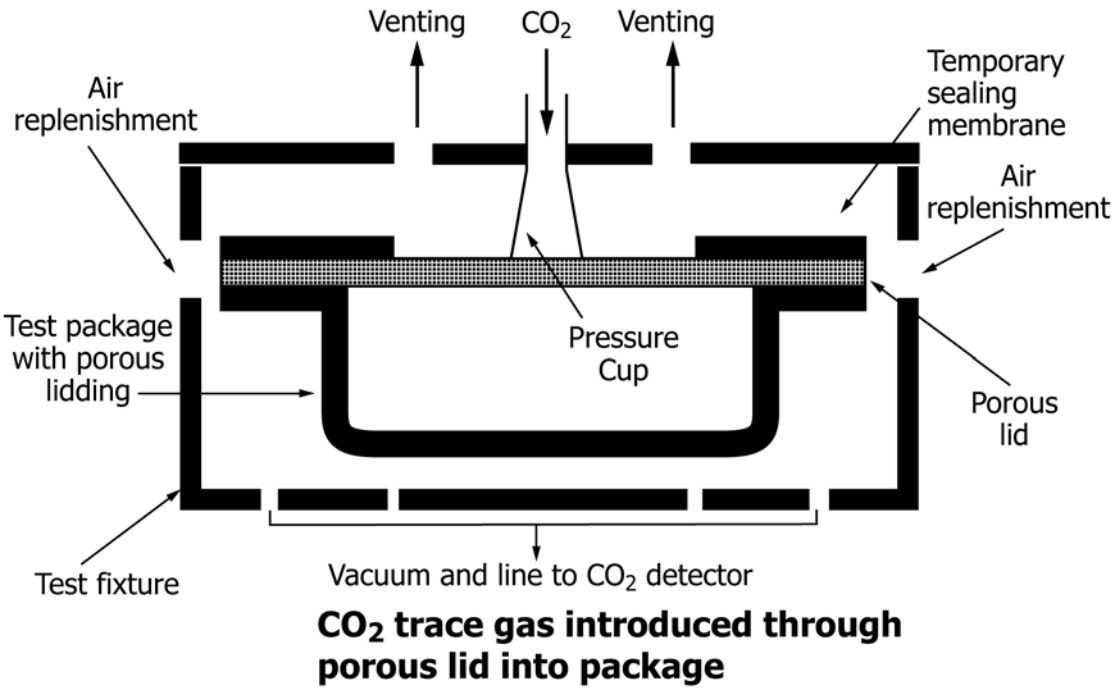

FIG. 1 Schematic of Test Fixture and Test Package

9.2 CO_2, although inert and non-toxic, can cause danger of suffocation if it is allowed to displace oxygen. Thus it is recommended that the spent carbon dioxide be naturally vented away from the test area and that adequate ventilation be provided.

10. Calibration and Standardization

10.1 Before any measurements are made, the apparatus must be calibrated. The calibration procedure is used for overall system checkout, as well as to establish an initial reference profile for a simulated channel or pinhole leak, and to determine test limits for each different package geometry that is to be tested using a specific test fixture. The calibration procedure is performed to establish the sensitivity setting of the instrumentation. It is expected that the calibration procedures are carried out frequently; typically, at least one or more times a day, preferably at the beginning of every shift.

10.2 Refer to the instrument manufacturer's operating instructions regarding preparation of Calibration Standards, Conditioning of Calibration Packages and Instrument Calibration used in establishing baseline settings.

11. Procedure

11.1 Verify that sufficient CO_2 trace gas is available for the tests. Check the trace gas supply and functionality of the gas delivery system.

11.2 Select and implement the properly sized test fixture for the packages to be tested. Verify that the instrument and associated test fixture have been calibrated for the packages to be tested. The test fixture is too large when the instrument is unable to detect a calibrated control channel or pinhole leak.

11.3 Adjust the instrument baseline settings determined in calibration.

11.4 Place the package assembly into the test fixture. Make certain that the package assembly is centered in the fixture and that the lidding side of the package is pointed up toward the test fixture hinged cover and incorporated sealing membrane.

NOTE 2—The sealing membrane needs to be clean in order to develop a good seal with the porous lidding of the package. Laboratory conditions may cause dust or debris to be collected on the sealing membrane. These conditions thus warrant frequent inspection and cleaning of the sealing membrane with a lint-free cloth soaked with a solvent recommended by the manufacturer of the equipment.

11.5 Close the top cover of the test fixture and make certain that the CO_2 port makes good contact with the package assembly.

11.6 Start the test.

11.7 Note the pass or fail indicator and record results. Set aside any "failed/defective" package for further evaluation. Further evaluation should include re-testing of the package.

11.8 Select another package and repeat the testing process.

12. Report

12.1 The report shall include the following:

12.1.1 A statement indicating that the tests were performed in accordance with ASTM Standard F2228, except where noted.

12.1.2 The serial numbers, calibration values and most recent calibration dates for all calibration standards used.

12.1.3 Record the date, time, location, and identification of the apparatus and the operator.

12.1.4 Record the package type, size, material, product, and traceable identification numbers.

12.1.5 Record the leak rate reject set point as programmed into the apparatus.

12.1.6 Record the number of packages tested, and the number of failed packages.

12.1.7 Record the failed packages to be rejected by identifying either individual serial numbers or lot numbers.

12.1.8 Record the disposition of good packages as well as failed packages.

12.1.9 Copies of any software-generated data sheets, or reports produced during the testing.

13. Precision and Bias

13.1 *Precision*—A round robin study was conducted in 2002, which included three laboratories. Packages with and without channels in the seal area and pinholes in the trays were tested for leaks. The equipment used in this interlaboratory study was the Pac Guard Model 500 available from MOCON.

13.1.1 Two different package sizes were tested. The larger package consisting of a PETE tray and an adhesive coated 1073B Tyvek lidding had the approximate outside dimensions of 129 mm wide by 167 mm long by 20 mm high (5.1 by 6.6 by 0.8 in.) with an internal volume of 208 mL. The smaller package consisted also of a PETE tray and an adhesive coated 1073B Tyvek lidding and had the approximate outside dimensions of 69 mm wide by 139 mm long by 18 mm high (2.7 by 5.5 by 0.7 in.) with an internal volume of 80.7 mL.

13.1.2 Three sample packages for each of two sizes (six packages) contained a 100 µm (0.004 in.) calibrated channel (leak), while six similar packages contained a 50 µm (0.002 in.) calibrated pinhole (leak), and six similar packages contained a plugged pinhole (no leak).

13.1.3 Each sample material was tested at three laboratories, using the same instrumentation. Each package was tested at the two possible orientations allowed by the instrument. Two other operators subsequently repeated the tests at different times. A grand total of 324 tests were performed, 216 on materials with leaks and 108 on materials without leaks.

13.2 Tables 1-3 represent a summary of the test data.

13.2.1 The results show that none of the 216 tests on calibrated leaks failed to detect the leak, that is, there were zero false negatives. Four of the 108 tests on plugged pinholes resulted in the determination of a leak, that is, a false positive.

13.2.2 Four errors occurred at Laboratory 2. It was observed that dust collected on the sealing membrane and thus proper sealing of the porous lidding may not have been accomplished. This condition would allow CO_2 to flow between the sealing

TABLE 1 Percent Incorrect by Material

Material	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
Package	324	4	1.2	3.1

TABLE 2 Percent Incorrect by Laboratory

Lab	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
1	108	0	0.0	3.4
2	108	4	3.7	9.2
3	108	0	0.0	3.4

TABLE 3 Percent Incorrect by Defect Type

Defect Type	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
Plugged pinhole	108	4	3.7	9.2
50 µm pinhole	108	0	0.0	3.4
100 µm channel	108	0	0.0	3.4
Open pinhole and channel combined	216	0	0.0	1.7

membrane and the package lidding into the test fixture where it would be detected and cause the alarm to be triggered. See Note 2 for inspection and cleaning guidelines for the sealing membrane.

13.2.3 Aside from laboratory conditions, it can be argued that the instrument tests were independent, and that the number of defects follows a binomial distribution with probability p of testing error. An estimate of p is the error rate, and a conservative confidence interval for p can be determined. The tables list the upper bound of a 95 % confidence interval for the true error rate.

13.2.4 Package size had no effect on the test results.

14. Keywords

14.1 basal flow; carbon dioxide (CO_2) leak testing; flexible packaging; infrared CO_2 sensor; medical packaging; non-destructive testing; package integrity monitoring; package integrity test; pass/fail criteria; pass/fail levels; permeable packaging; pinhole leaks; porous barrier; porous lids; porous packaging; rigid thermoformed trays; sealing membrane performance; seal integrity monitoring; seal integrity test; seal leaks; sterile integrity tests; trace gas leak testing

ANNEX

(Mandatory Information)

A1. LEAK TEST THEORY

A1.1 Placing the lidded package in a test fixture to which the infrared sensor is connected initiates the test method. This method does not locate the leak, but instead provides a measurement of total leakage. The response of the instrument to any leakage is a non-linear function of the leak (hole) size. The sensitivity of this method is a function of trace gas concentration in the package. It is recommended that the trace gas delivery flow be adjusted to a rate that equals at least two or more tray volume exchanges per test cycle time, thus yielding close to 90 to 100 % trace gas concentration levels within the package.

A1.2 In this non-destructive test procedure, leaks in medical packages, rigid thermoformed trays with porous lids, are detected. As shown in Fig. 1, the package with porous lidding is placed in a test fixture where CO_2 trace gas is delivered to the outside of the porous lid material through a port in the sealing membrane. The trace gas is forced under pressure through the porous lidding into the cavity of the package. Venting, of the excess gas mixture from the package, away from the test fixture is provided.

A1.3 Upon initiation of the test cycle, the package is flushed with CO_2 trace gas at a predetermined flow rate. The internal package pressure of CO_2 is generally set between 0.25 and 0.75 kPa (0.0363 to 0.1088 psi or 1 to 3 in. of water H_2O column). During this flushing/soaking cycle, room air is drawn through the test fixture at a low vacuum of approximately 0.0249 kPa (0.00361 psi or 0.1 in. of H_2O column) and channeled past the infrared detection sensor. Near the end of the flushing/soaking cycle, a solenoid valve is energized causing the room air drawn flow to be cut off, thus producing an ambient pressure within the capture volume of the test fixture. If the package has a seal channel leak, pinhole or crack then some carbon dioxide will leak out of the package and accumulate in the capture volume of the test fixture.

A1.4 At the end of the test period, the solenoid valve reverts back to its initial (open) position. This permits room air to again flow to the test fixture chamber. A pump then flushes the room air through the capture volume, which in turn picks up any CO_2 that may have escaped out of the package and pulls it through the infrared sensing chamber. If the carbon dioxide concentration in the capture volume is significantly higher than that of the room air, the carbon dioxide alarm will be activated. In order to reduce potential false positive signal responses from the instrumentation, it is recommended that the ambient carbon dioxide levels not fluctuate since varying levels of carbon dioxide may affect the sensitivity of the system. Thus, it is highly recommended that the exhausted CO_2 be naturally vented, through appropriate piping, away from the test area.