



Designation: F2228 – 13 (Reapproved 2021)

Standard Test Method for Non-Destructive Detection of Leaks in Packaging Which Incorporates Porous Barrier Material by CO₂ Tracer Gas Method¹

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. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D996 Terminology of Packaging and Distribution Environments

¹ This test method is under the jurisdiction of ASTM Committee F02 on Primary 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.

F17 Terminology Relating to Primary 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, n*—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, n*—a compound selected solely for use to identify leakage flow.

4. Summary of Test Method

4.1 This test method utilizes CO₂ sensing techniques in the detection of a CO₂ 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.

³ The last approved version of this historical standard is referenced on www.astm.org.

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 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 *CO₂ 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.

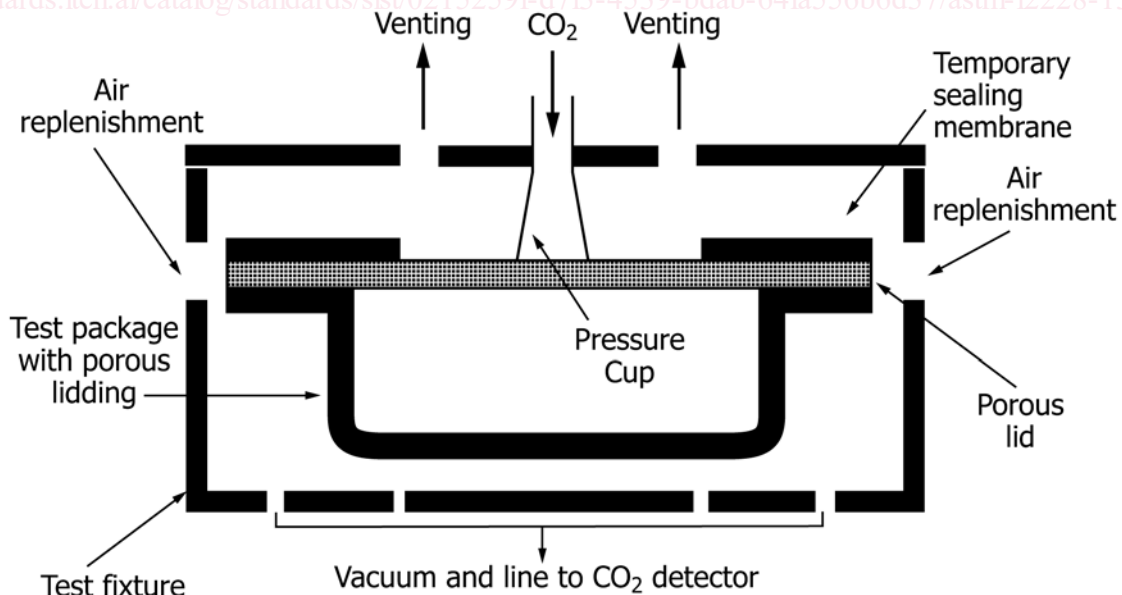
8.3.4 Damage to the printed information on the porous barrier. The adhesive of the sealing membrane may lift off the ink from the barrier.

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.



CO₂ trace gas introduced through porous lid into package

FIG. 1 Schematic of Test Fixture and Test Package

9.2 CO₂, 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 CO₂ 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 CO₂ 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 CO₂ 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