Designation: F2227 – 13

Standard Test Method for Non-Destructive Detection of Leaks in Non-sealed and Empty Packaging Trays by CO_2 Tracer Gas Method[1]

This standard is issued under the fixed designation F2227; 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 pinhole leaks in trays, as small as 50 µm (0.002 in.) in diameter, or equivalently sized cracks, subject to trace gas concentration in the tray, tray design and manufacturing tolerances.

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

F1327 Terminology Relating to Barrier Materials for Medical Packaging (Withdrawn 2007)[3]

3. Terminology

3.1 *General Term Definitions*—For definitions used in this test method, see Terminologies D996 and F1327, Sections 3.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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 trays. The test method provides a qualitative (accept/reject) inspection method to evaluate trays for pinholes and cracks. 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 imperfections such as pinholes or cracks in trays.

5.2 After initial instrument set-up and calibration, the operations of individual tests and test results do not need operator interpretation.

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 and 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 membrane is to seal off the tracer gas transmission out of the top of the open tray.

6.3 *Control Trays*—Calibrated pinholes, or leaks, constructed in control trays 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.

[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.

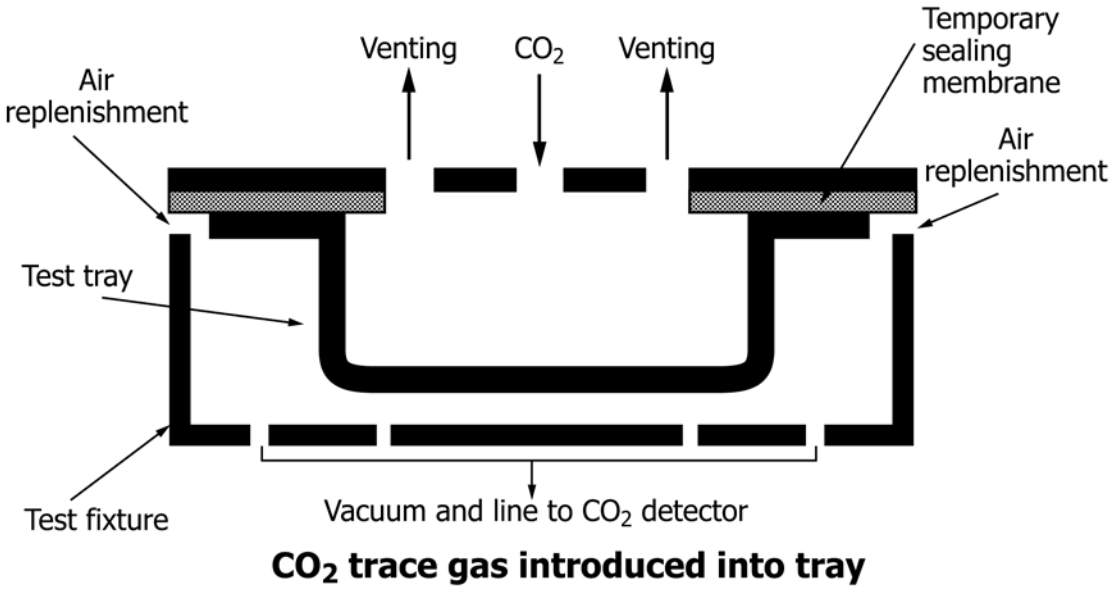

FIG. 1 Schematic of Test Fixture and Test Tray

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 sealing membrane must exhibit the correct pliability and tackiness in order to form a gas-tight bond without leaving a residue on the tray-sealing surface after removal from the test fixture.

9. Hazards

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

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 simulated pinhole leaks, and to determine test limits for each different tray geometry 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 be 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 Trays and Instrument Calibration used in establishing baseline settings.

11. Procedure

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

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

11.3 Adjust the instrument baseline settings determined in calibration.

11.4 Place the tray to be tested into the test fixture making certain that the tray is centered in the fixture and that good sealing contact is made between the tray flange and the fixture incorporated sealing.

NOTE 1—The sealing membrane needs to be clean in order to develop a good seal with the sealing flange of the tray. Laboratory conditions may cause dust or debris to be collected on the sealing membrane. These conditions thus will 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.

11.6 Start the test.

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

11.8 Select another tray 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 F2227, 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 tray 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 trays tested, and the number of failed trays.

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

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

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. Trays with and without pinhole leaks 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 tray sizes were tested. The large PETE trays 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 small PETE trays 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 trays for each of two sizes (six trays) had a 50 µm (0.002 in.) calibrated pinhole (leak), and six similar trays had a plugged pinhole (no leak).

13.1.3 Each sample material was tested at three laboratories, using the same instrumentation. Each tray 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 216 tests were performed, 108 on materials with leaks and 108 on materials without leaks.

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

TABLE 1 Percent Incorrect by Material

Material	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
Tray	216	0	0.0	1.7

TABLE 2 Percent Incorrect by Laboratory

Lab	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
1	72	0	0.0	5.0
2	72	0	0.0	5.0
3	72	0	0.0	5.0

TABLE 3 Percent Incorrect by Defect Type

Defect Type	Test Determinations	Incorrect Analysis	Percent (%) Incorrect	95 % Upper Bound
Plugged pinhole	108	0	0.0	3.4
50 µm pinhole	108	0	0.0	3.4

13.2.1 The results show that none of the 108 tests on calibrated leaks failed to detect the leak, that is, there were zero false negatives. There were also no false positives.

13.2.2 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.

13.2.3 Tray size had no effect on the test results.

13.3 *Bias*—The bias for this test method has not been determined because there is no known standard reference available.

14. Keywords

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

ANNEX

(Mandatory Information)

A1. LEAK TEST THEORY

A1.1 Placing the unlidded tray 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 tray. 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 tray.

A1.2 In this test procedure, pinhole leaks or cracks in trays are detected. As shown in Fig. 1, the tray is placed in a test fixture such that a good, vacuum tight contact is made between the tray flange and associated fixture sealing (sealing membrane). CO_2 trace gas is then introduced into the sealed cavity of the tray.

A1.3 Upon initiation of the test cycle, the tray is flushed with CO_2 trace gas at a predetermined flow rate. The internal tray 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 tray has a pinhole or crack then some carbon dioxide will leak out of the tray 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 tray 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.