Designation: F 2338 – 05

Standard Test Method for Nondestructive Detection of Leaks in Packages by Vacuum Decay Method[1]

This standard is issued under the fixed designation F 2338; 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 *Test Packages*—Packages that can be nondestructively evaluated by this test method include:

1.1.1 Rigid and semi-rigid non-lidded trays.

1.1.2 Trays or cups sealed with porous barrier lidding material.

1.1.3 Rigid, nonporous packages.

1.1.4 Flexible, nonporous packages (see 1.2.4).

1.2 *Leaks Detected*—This test method is capable of detecting package leaks using an absolute or differential pressure transducer leak detector. The sensitivity of a test is a function of the sensitivity of the transducer, the package design, the design of the package test fixture, and critical test parameters of time and pressure. Types and sizes of leaks that may be detected for various package systems, as well as test sensitivities are described below. These data are based on precision and bias confirmation studies.

1.2.1 *Trays or Cups (Non-lidded)*—Hole or crack defects in the wall of the tray/cup of at least 50 µm in diameter can be detected at a Target Vacuum of $4{\cdot}10^4$ Pa (400 mbar) using an absolute pressure transducer test instrument.

1.2.2 *Trays Sealed with Porous Barrier Lidding Material*—Hole or crack defects in the wall of the tray/cup of at least 100 µm in diameter can be detected. Channel defects in the seal area (made using wires of 125 µm in diameter) can be detected. Severe seal bonding defects in both continuous adhesive and dot matrix adhesive package systems can be detected. Slightly incomplete dot matrix adhesive bonding defects can also be detected. All porous barrier lidding material packages were tested at a Target Vacuum of $4{\cdot}10^4$ Pa (400 mbar) using an absolute pressure transducer test instrument. Using a calibrated volumetric airflow meter, the sensitivity of the test for porous lidded packages is shown to be approximately 10^{-2} $Pa{\cdot}m^3{\cdot}s^{-1}$.

1.2.3 *Rigid, Nonporous Packages*—Hole defects of at least 5 µm in diameter can be detected. All rigid, nonporous packages were tested at a target vacuum of $5{\cdot}10^4$ Pa (500 mbar) using a differential pressure transducer test instrument. Using a calibrated volumetric airflow meter, the sensitivity of the test for rigid, nonporous packages is shown to be approximately 10^{-4} $Pa{\cdot}m^3{\cdot}s^{-1}$.

1.2.4 *Flexible, Nonporous Packages*—Such packages may also be tested by the vacuum decay method using either an absolute or differential pressure tranducer test instrument. The instrument should be selected based on the leak test sensitivity desired. Sensitivity data for flexible packages were not included in the precision and bias studies, although the use of vacuum decay for testing such packages is well known.

1.3 *Test Results*—The test results are qualitative (Accept/Reject). Acceptance criteria for test results are established from quantitative baseline vacuum decay measurements obtained from control, non-leaking packages.

1.4 *Standard Value Units*—The values used in this test method are stated in SI units and are to be regarded as standard units. Values 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]

D 996 Terminology of Packaging and Distribution Environments

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F 17 Terminology Relating to Flexible Barrier Materials

F 1327 Terminology Relating to Barrier Materials for Medical Packaging

3. Terminology

3.1 *Definitions*—For definitions used in this test method, see Terminologies D 996, F 17, and F 1327.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials 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.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *baseline vacuum decay*, *n*—the extent of vacuum change within the test chamber over time demonstrated by a control, non-leaking package.

3.2.2 *control, non-leaking packages*, *n*—packages without defects and properly sealed or closed according to manufacturer's specifications.

3.2.3 *flexible, nonporous packages*, *n*—packages that significantly deflect when under vacuum, and are constructed of malleable, nonporous materials. Examples include pouches or bags made of polymeric, foil, or laminate films.

3.2.4 *rigid, nonporous packages*, *n*—packages that do not significantly deflect under vacuum and are constructed of solid, nonporous materials. For example, plastic bottles with screw-thread or snap-on closures are rigid, nonporous packages.

3.2.5 *semi-rigid trays or cups*, *n*—trays made of material that retain shape upon deflection. For example, thermoformed PETE or PETG trays are considered semi-rigid trays.

3.2.6 *spotty or mottled seals*, *n*—an incomplete adhesive bond made between a package tray or cup and porous lidding material that can be visibly identified by a distinctive pattern of dots, spotting or mottling on the tray sealing surface after the lid is removed.

3.2.7 *volumetric airflow meter*, *n*—a calibration tool that can be used to provide an artificial leak of known volumetric airflow rate into the test chamber for verification of instrument sensitivity. Airflow meters should be calibrated to NIST standards. The operational range of the meter should reflect the desired limit of sensitivity for the intended leak test.

3.3 *Definitions of Test Cycle and Critical Parameters Terms*—For terms and abbreviations relating to the test cycle and the critical parameters for establishing accept/reject limits, see Annex A1.

4. Summary of Test Method

4.1 The test package is placed in a test chamber to which vacuum is applied. The chamber is then isolated from the vacuum source and an absolute or differential vacuum transducer is used to monitor the test chamber for both the level of vacuum, as well as the change in vacuum over time. Vacuum decay, or rise in chamber pressure, is a result of package headspace gas being drawn out of the package through any leaks present, plus background noise. Leak detection requires vacuum decay in excess of the background noise level. Background noise vacuum decay may result from package expansion when exposed to vacuum (flexible or semi-rigid packages), or from residual gases inherent in the test chamber or test system lines.

4.2 Porous barrier lidded tray or cup packages are tested for leaks located in the tray or cup, and at the lidding material/tray seal junction. Leaks in the porous lidding material itself cannot be detected. When testing such packages, steps are taken to physically mask or block the porous barrier surface to prevent the migration of package gas through the porous lid. These steps may require some sample preparation, depending on the masking approach required, but must be nondestructive and noninvasive. Vacuum decay from porous barrier lidded packages may potentially include background noise from gas trapped between the lidding material and the masking surface, or from transverse gas flow through the porous barrier material itself at the lid/tray seal junction.

4.3 The sensitivity of a vacuum decay leak test is a function of several factors. Smaller leaks can be detected with more sensitive pressure transducers, and with longer test times. Also, pressure changes can be more readily detected with smaller void volumes between the test package and the test chamber, and with smaller test system line volumes. Steps to reduce background noise can also improve sensitivity. For example, for porous barrier lidded packages, more effective masking techniques will minimize background noise.

NOTE 1—Further information on the "Leak Test Theory" may be found in Annex A1.

5. Significance and Use

5.1 Leaks in medical device, pharmaceutical and food packages may result in the ingress of unwanted gases (most commonly oxygen), harmful microbiological or particulate contaminants. Package leaks may appear as imperfections in the package components themselves or at the seal juncture between mated components. The ability to detect leaks is necessary to ensure consistency and integrity of packages.

5.2 After initial set-up and calibration, the operations of individual tests may be semi-automatic, automatic or manual. The test method permits the non-destructive detection of leaks not visibly detectable. The test method does not require the introduction of any extraneous materials or substances, such as dyes or gases. However, it is important to physically mask or block off any porous barrier surface of the package during the test to prevent a rapid loss of chamber vacuum resulting primarily from gas migration through the porous surface. Leak detection is based solely on the ability to detect the change in pressure inside the test chamber as a result of air egress from the properly masked package when challenged with vacuum conditions.

5.3 This test is a useful research tool for optimization of package sealing parameters and for comparative evaluation of various packages and materials. This test method is also applicable to production settings as it is rapid, non-invasive and non-destructive, making it useful for either 100 % on-line testing or to perform tests on a statistical sampling from the production operation.

5.4 Leak test results that exceed the permissible limits for the vacuum decay test are indicated by audible or visual signal responses, or both.

6. Apparatus

6.1 *Vacuum Decay Leak Detection Apparatus*—All vacuum decay test systems include a test chamber with a lower compartment (lower tooling) designed to nest the test package, and an upper lid (top tooling) for closing the test chamber. Fig. 1 illustrates a test chamber designed for testing packages with porous barrier lidding material. The test fixture upper lid consists of a flexible bladder to mask the package's porous barrier during the test cycle. Fig. 2 illustrates a test chamber designed for testing rigid, nonporous packages. In this case, there is no flexible bladder. For both test chamber designs, the test chamber is connected to the vacuum decay test system. This system includes a vacuum source for establishing vacuum

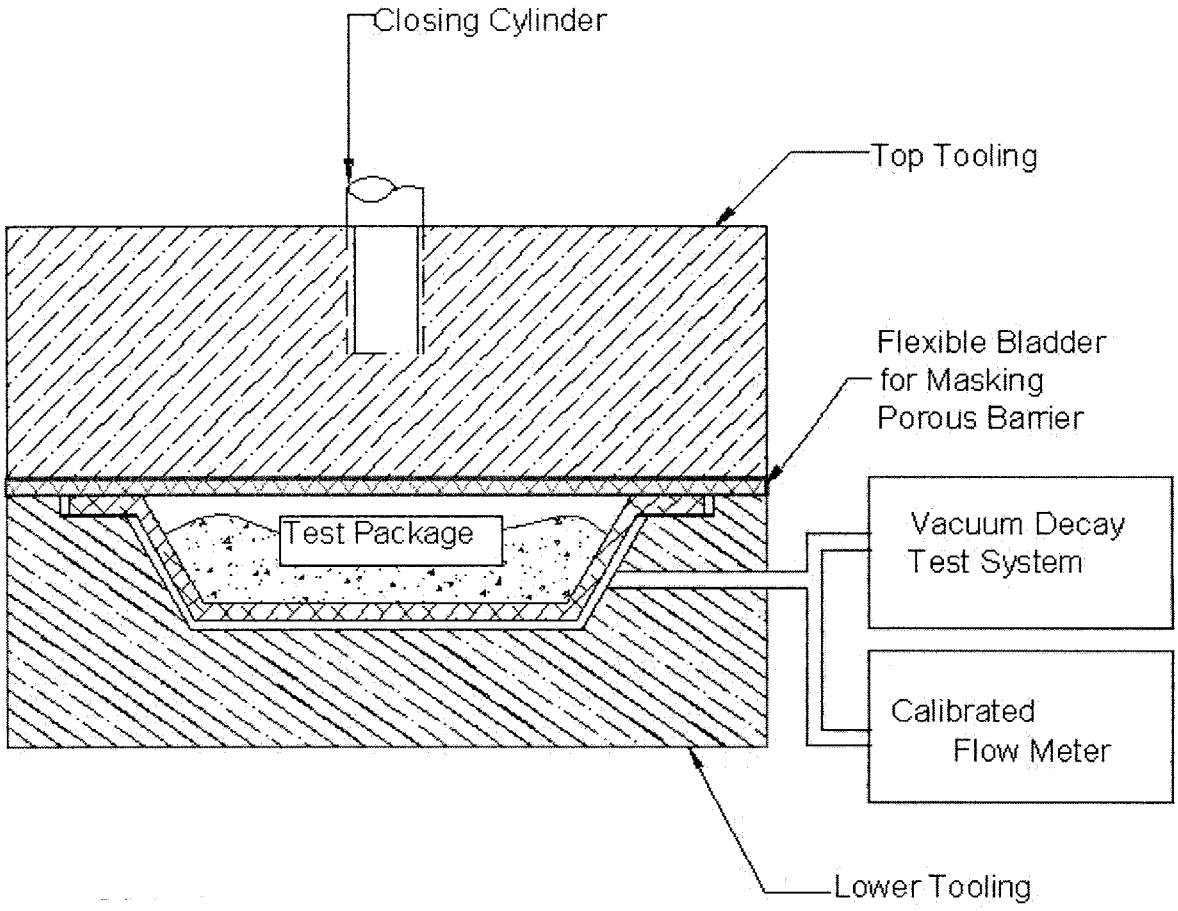

FIG. 1 Schematic of Fixture and Porous Barrier Lidded Test Package

within the chamber at the beginning of the test cycle, and an absolute or differential pressure transducer for monitoring the level of vacuum as well as the pressure change as a function of time during the test cycle. A calibrated volumetric airflow meter may be placed in-line with the test system for verifying the sensitivity of a leak test.

6.2 *Tray Nest or Lower Tooling*—The bottom half of the test chamber is dimensionally designed to closely nest the test package, while still allowing for easy gas flow around the test package. Without ready gas flow around the package, leakage sites can be blocked. Conversely, the larger the gap between the test chamber and the test package, the less sensitive the leak test, as vacuum decay from package leakage will be minimized in a larger net test chamber volume.

6.3 *Upper Lid or Upper Tooling*—The upper lid is designed to tightly seal the closed test chamber during the vacuum cycle.

6.4 *Mask or Block*—The porous barrier lidding material of packages must be masked or blocked during testing to minimize egress of air from the package through the lidding. Various masking techniques may be used, including a test chamber designed with a flexible bladder in the upper tooling (refer to Fig. 1).

6.5 *Volumetric Airflow Meter*—An adjustable volumetric airflow meter is placed in-line with the test chamber to introduce an artificial leak of variable size. It is recommended that an airflow meter be used to verify the sensitivity of the leak test parameters.

NOTE 2—Refer to Annex A2 for further information about the use of a volumetric airflow meter for verifying leak test sensitivity.

7. Hazards

7.1 As the test chamber is closed, it may present pinch-point hazards.

8. Preparation of Apparatus

8.1 The test apparatus must be started, warmed-up, and made ready according to the manufacturer's specifications. Utilities required for instrument operation include electrical power and a supply of dry, non-lubricated compressed air, according to manufacturer's specifications.

9. Calibration and Standardization

9.1 Before test measurements are made, the apparatus must be calibrated. The pressure transducers, the vacuum source pressure gage, and the adjustable volumetric airflow meter must all be calibrated according to the manufacturer's recommended procedures and maintenance schedule.

9.2 Critical test parameter settings must be established for each package/test fixture combination. Parameters will vary based on the test package geometry and any porous barrier surface's inherent porosity.

NOTE 3—Refer to Section 4 and Annex A1 for a description of critical test parameters.

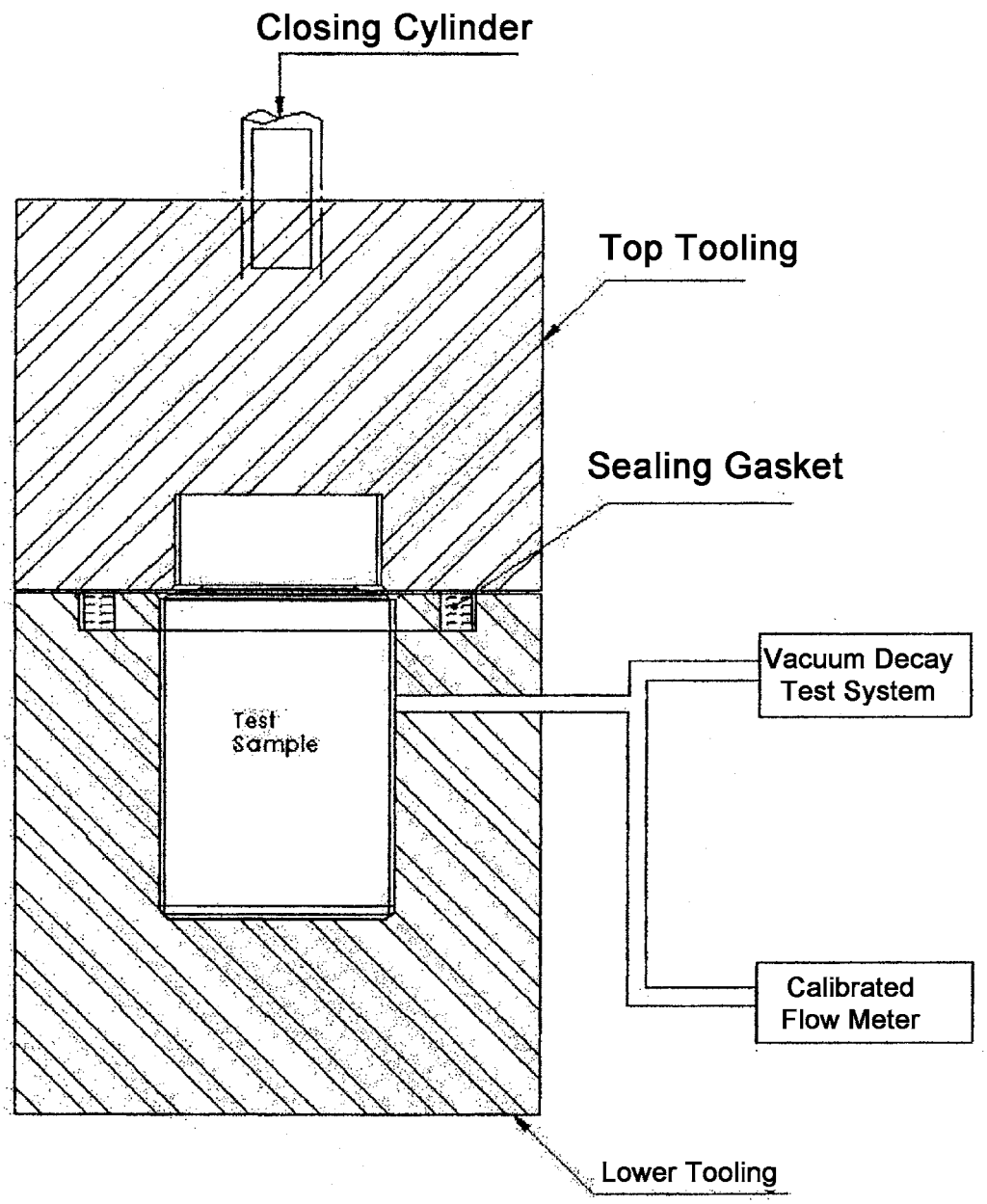

FIG. 2 Schematic of Fixture and Rigid, Nonporous Test Package

9.3 A sample population of control non-leaking packages must be used for selecting and optimizing critical test parameters. Control packages are to be made from the same materials and according to the same design as the test units.

NOTE 4—Refer to Annex A2 for information on critical test parameter selection.

9.4 After critical test parameters have been selected, qualify the ability of the test to reliably differentiate between known non-leaking and defective packages.

9.5 Determine the sensitivity of the test using control non-leaking test packages and a calibrated volumetric airflow meter.

NOTE 5—Refer to Annex A2 for information about test sensitivity verification procedures.

9.6 Test qualification (see 9.4) and test sensitivity verification (see 9.5) are to be conducted frequently, typically at least one or more times a day, preferably at the beginning of every shift.

10. Procedure

10.1 Select and install the appropriately sized test chamber for the package to be tested. Make any necessary adjustments to the chamber to ensure a sufficiently tight seal of the chamber lid (upper tooling) to the lower chamber package nest (lower tooling) when the test chamber is in the closed position.

TABLE 1 Nonlidded Tray Leak Detection Results

Approximate Tray Size (cm) L × W × H	Tray Description	Number of Units Tested	Total Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	Success Rate (% accurate replicate tests)
14 × 7 × 2	No defect	5	45	0	45	100
	100 µm hole	4	36	36	0	100
17 × 13 × 2	No defect	5	45	0	45	100
	50 µm hole	5	45	35	10[A]	78 (100)[A]
	100 µm hole	5	45	45	0	100

[A] Two test packages yielded all 10 PASS observations. An independent test laboratory later verified that the holes in these packages could no longer be located and may have become clogged. In this case, the success rate is reported considering all 5 test trays (78 %), and considering only the 3 known defective trays (100 %).

10.2 Verify the pressure level available at the supply source. Check the functionality of the vacuum source.

10.3 Program the test instrument with all necessary test parameters and accept/reject criteria.

10.4 Place the assembled package into the lower tooling nest and close the test chamber. Take appropriate steps to mask or block any porous barrier surface of the package.

NOTE 6—Inspect and clean the masking or blocking surface according to a regularly established routine according to the instrument manufacturer's recommended procedures to ensure effective masking of the porous barrier surface.

10.5 Start the test.

10.6 Note the pass or fail indicator or other means of detecting vacuum decay and document results. Identify and set aside any failed package for further evaluation.

10.7 Select another package and repeat the testing process.

11. Report

11.1 For each package tested, report the values for the following critical test parameters as well as package test results:

11.1.1 Reserve Vacuum (VRes),

11.1.2 Target or Test Vacuum (Vac),

11.1.3 Reference Vacuum (VRef),

11.1.4 Reference Fill Time (TfillRef),

11.1.5 Equalization Time (Tequal),

11.1.6 Vacuum Decay Test Time (Ttest),

11.1.7 Reference Vacuum Decay Rate Accept/Reject Limit (dP/dt Ref), and

11.1.8 Accept/Reject Test Results.

NOTE 7—Refer to Annex A1 for definitions of critical test parameters.

12. Precision and Bias

12.1 *Nonlidded and Porous Barrier Lidded Trays*—An interlaboratory study was run in accordance with Practice E 691 using an absolute pressure decay instrument.[3] Three laboratories ran the study, each using a separate instrument. Each laboratory performed three replicate tests on each test sample. Test sample populations consisted of non-lidded semi-rigid (PETE) thermoformed trays, and trays sealed with various porous barrier lidding materials. The same test samples were tested at each laboratory.[4] Test results are qualitative in nature (Pass or Fail). Operators selected test critical parameters for each sample population; therefore test results reflect operator, laboratory and instrument variability.

12.1.1 *Nonlidded Trays*—As summarized in Table 1, two populations of non-lidded trays representing two tray sizes were tested. Defective samples contained a single hole in the tray wall of either 50 µm or 100 µm in diameter. Two of the five larger trays, each with a 50 µm hole, repeatedly failed to be detected at more than one test site, while the other three trays were consistently identified as leaking. At the completion of the study the two suspect trays were independently reexamined for the presence and size of the holes. It was determined that the holes could no longer be located and it was hypothesized that they had become clogged. Eliminating the two suspect trays, results demonstrate the ability of the test method to identify defective trays with holes ≥50 µm, when using a Target Vacuum (Vac) of $4 \cdot 10^4$ Pa (400 mbar).

12.1.2 *Porous Barrier Lidded Trays*—As per the results outlined in Table 2, two populations of porous barrier lidded tray packages were tested, representing two package sizes, all sealed with one type of coated porous barrier lidding material. Defective samples included packages with a single hole in the tray wall (50 µm or 100 µm in diameter), and packages with a single seal channel defect created using a wire of either 75 µm, 100 µm, or 125 µm in diameter (0.003, 0.004, and 0.005 in., respectively). An independent laboratory microscopically verified tray hole sizes, however seal channel sizes could not be reliably verified. Results demonstrate the ability of the test method to identify defective packages sealed with porous barrier lidding material. Defects consistently identified include tray holes of at least 100 µm in diameter, and channel defects created using a 125 µm wire, when using a Target Vacuum of $4 \cdot 10^4$ Pa (400 mbar).

12.1.3 *Porous Barrier Lidded Trays with Various Adhesive Bonding Systems*—Table 3 documents test results using two populations of tray packages with porous barrier lidding material representing two seal bonding adhesive systems. All lidding materials consisted of the same porous barrier substrate. Adhesives included dot matrix (C) and continuous (D) systems. Defective samples with incomplete seal bonding were included. For dot matrix adhesive seals, defect severity was visually judged at the independent laboratory where the packages were sealed. Continuous adhesive seals could not be visually verified with accuracy; therefore, only sealing conditions were used to classify packages. Results demonstrate the ability of the test method to reliably identify packages with less than optimum seal bonding for dot matrix adhesive systems, at a Target Vacuum (Vac) of $4 \cdot 10^4$ Pa (400 mbar). Severely

[3] Model Pti 225 by Packaging Technologies and Inspection, 145 Main Street, Tuckahoe, NY 10707.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: F02–1019.

TABLE 2 Trays with Porous Barrier Lidding Leak Detection Results

Approximate Package Size (L × W × H)	Porous Barrier Lidding Material	Package Description	Number of Package Units Tested	Total Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	Success Rate (% accurate replicate tests)
196 cm^3 (14 × 7 × 2)	A	No defect	5	45	2	43	96
		50 µm hole	5	45	36	9	80
		100 µm hole	5	45	45	0	100
		Channel made with 75 µm wire	5	45	15	30	33
		Channel made with 100 µm wire	5	45	45	0	100
		Channel made with 125 µm wire	5	45	45	0	100
536 cm^3 (16.5 × 13 × 2.5)	A	No defect	5	45	0	45	100
		50 µm hole	5	45	16	29	36
		100 µm hole	5	45	45	0	100
		Channel made with 75 µm wire	5	45	1	44	2
		Channel made with 100 µm wire	5	45	40	5	89
		Channel made with 125 µm wire	5	45	45	0	100

TABLE 3 Trays with Porous Barrier Lidding Seal Bonding Defect Detection Results

Approximate Package Size (L × W × H)	Porous Barrier Lidding Material	Bonding Adhesive[A]	Package Description	Number of Package Units Tested	Total Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	Success Rate (% accurate replicate tests)
536 cm^3 (16.5 × 13 × 2.5)	A	C	No defect	5	45	0	45	100
			Slightly incomplete bonding	5	45	45	0	100
			Severely incomplete bonding	5	45	45	0	100
536 cm^3 (16.5 × 13 × 2.5)	A	D	No defect	5	45	0	45	100
			Slightly incomplete bonding	5	45	32	13	71
			Severely incomplete bonding	5	45	45	0	100

[A] Bonding adhesives were either continuous (D) or dot matrix (C) adhesive systems.

incomplete bonds made with continuous adhesive systems could be detected at a Target Vacuum (Vac) of $4 \cdot 10^4$ Pa (400 mbar).

12.2 *Trays with Various Porous Barrier Lidding Materials*—Table 4 summarizes a single laboratory study run using a single absolute pressure decay instrument[3] to verify the test method's feasibility in evaluating semi-rigid thermoformed tray packages sealed with various types of porous barrier lidding materials, and to obtain an estimate of the tests' sensitivity.[4]Critical test parameters were identified for each package population. The sensitivity of each test was determined by introducing a controlled flow of air using a calibrated volumetric airflow meter into the instrument test chamber containing the test package. The sensitivity of the test was defined as the leak rate that first triggered FAIL test results. Results demonstrate that the test method can be used to test packages sealed with various types of porous barrier lidding material, and that the tests are similar in sensitivity (approximately 10^{-2} $Pa \cdot m^3 \cdot s^{-1}$ at a Target Vacuum [Vac] of $4 \cdot 10^4$ Pa [400 mbar]).

12.3 *Rigid, Nonporous Packages*—A single laboratory study was run using three differential pressure decay instruments[5] to verify the test method's feasibility in evaluating rigid, nonporous packages, and to obtain an estimate of the tests' sensitivity (see ASTM Research Report No. F02–1020[6]). The packages tested included two sizes of plastic (HDPE) bottles, 30-mL and 75-mL capacity, sealed with induction seals, and capped with non-child-resistant screw-thread caps (for the 30-mL bottles) and child-resistant push-and-turn screw-thread caps (for the 75-mL bottles). Defective packages were made by the introduction of a single laser-drilled hole in the induction seal face (including holes <5, 5, 10, 25 and 50 microns in diameter). After defects were created, caps were torqued onto the bottles. Three replicate tests were performed on each test sample using each test instrument.

12.3.1 Results summarized in Table 5 demonstrate the ability of the test method to identify defective packages with holes at least 5 µm in diameter, with a high probability of detecting hole sizes even smaller than 5 µm, when using a Target Vacuum (Vac) of $5 \cdot 10^4$ Pa (500 mbar). Holes rated as less than 5 µm could not be reliably sized microscopically. No control packages were falsely rejected.

12.3.2 The same Critical Test Parameters were utilized for all three test units, for all test sample populations. The sensitivity of each test was determined by introducing a controlled flow of air using a calibrated volumetric airflow meter into the instrument test chamber containing a series of control test packages. The sensitivity of the test was defined as the leak rate that first triggered FAIL test results. In every case, the test method was capable of detecting a gas flow-meter rate set to deliver a flow of 0.26 ccm. Actual measured gas flow meter readings ranged from 0.25 to 0.27 ccm for all control, pilot samples tested (equivalent to 4.2 to 4.5 $\cdot 10^{-4}$.$Pa \cdot m^3 \cdot s^{-1}$).

12.4 *Flexible, Nonporous Packages*—No precision and bias studies have been generated for inclusion in this method, although the use of vacuum decay leak testing for such packages is well known.

13. Keywords

13.1 absolute pressure transducer; barrier performance; block; criteria parameters; differential pressure transducer;

[5] Model Pti 325 by Packaging Technologies and Inspection, 145 Main Street, Tuckahoe, NY 10707.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: F02–1020.

TABLE 4 Leak Test Sensitivity Results—Control, No Defect Packages with Various Porous Barrier Lidding Materials

Approximate Package Size (L × W × H)	Porous Barrier Lidding Material	Bonding Adhesive	Leak Rate Introduced $cm^3 \cdot min^{-1}$	Leak Rate Introduced $Pa \cdot m^3 \cdot s^{-1}$	Number of Tests Performed	FAIL (Leak detected)	PASS (No leak detected)	Success Rate (% accurate tests)
536 cm^3	A	C	0	0	15	0	15	100
(16.5 × 13 × 2.5)			26	$4 \cdot 10^{-2}$	4	1	3	25
			29 to 52	(5 to 9) 10^{-2}	16	16	0	100
536 cm^3	A	D	0	0	15	0	15	100
(16.5 × 13 × 2.5)			17	$3 \cdot 10^{-2}$	5	2	3	40
			19 to 35	(3 to 6) 10^{-2}	15	15	0	100
536 cm^3	B	E	0	0	4	0	4	100
(16.5 × 13 × 2.5)			13 to 21	(2 to 3) 10^{-2}	6	3	3	50
			22 to 34	(4 to 6) 10^{-2}	6	6	0	100

NOTE—The simulated leak flowmeter is programmed to display units of $cm^3 \cdot min^{-1}$ (ccm); conversions to equivalent SI units of $Pa \cdot m^3 \cdot s^{-1}$ are also given.

TABLE 5 Leak Test Results—Rigid, Nonporous HDPE Bottles with Induction Seals and Screw-Thread Closures

Package	Defect Description	Number of Package Units Tested	Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	% Results Accurate
30–mL, Induction Seal,	No defect	35	315	0	315	100
Screw-thread	< 5 µm hole	3	27	25	2	93
Non-Child-Resistant	5 µm hole	3	27	27	0	100
Closure	10 µm hole	3	27	27	0	100
	25 µm hole	3	27	27	0	100
	50 µm hole	3	27	27	0	100
75–mL, Induction Seal,	No defect	35	315	0	315	100
Screw-thread	< 5 µm hole	3	27	26	1	96
Push and Turn	5 µm hole	3	27	27	0	100
Child-Resistant Closure	10 µm hole	3	27	27	0	100
	25 µm hole	3	27	27	0	100
	50 µm hole	3	27	27	0	100

flexible packaging; food packages; hole leaks; holes; leaks; mask; medical packages; non-destructive testing; package integrity monitoring; package integrity test; permeable packaging; pharmaceutical packages; porous barrier; porous barrier material; porous packaging; pressure change leak testing; pressure transducer; seal integrity monitoring; seal integrity test; seal leaks; semi-rigid thermoformed trays; sterile integrity test; vacuum decay leak testing; vacuum leak testing; volumetric airflow meter

ANNEXES

(Mandatory Information)

A1. VACUUM DECAY LEAK TEST THEORY

A1.1 A vacuum decay leak test procedure works on the principle that all leakage passageways will allow for gas migration out of a package when the package is exposed to external vacuum conditions. The rise in test chamber pressure during a test cycle, as monitored by a pressure transducer, is the result of internal package headspace gases migrating out of leaks in the package, plus background noise. Leak detection requires vacuum decay in excess of the background noise level. Background noise vacuum decay may result from package expansion when exposed to vacuum, or from trace gas in the test chamber or test system lines.

A1.2 Packages that include a porous barrier lidding material can be tested after physically masking or blocking off the package's porous barrier surface to minimize the amount of gas that is able to move out of the package through the porous barrier. Porous barrier lidding defects cannot be detected, however, defects in the seal area or in the tray itself can be. Vacuum decay from porous barrier lidded packages may potentially include background noise from gas trapped between the lidding material and the masking surface, or from transverse gas flow through the porous barrier material itself at the lid/tray seal junction.

A1.3 A typical test cycle consists of first placing the test package inside the test chamber and masking or blocking any porous barrier surface. Vacuum is drawn in the closed test chamber. At the end of the predetermined time period allowed for attaining initial target vacuum, the test chamber is isolated from the vacuum source. After a short time period allowed for equalization, the vacuum level and the rate of vacuum decay in the test chamber are monitored over a predetermined test time. For many packages, the time from chamber closing to completion of the test cycle may be as short as a few seconds. The

various critical test parameters of time and pressure for a test cycle are described below.

A1.3.1 *Reserve Vacuum (VRes)*—Reserve Vacuum should be set to approximately 10 % greater than Target Vacuum. Reserve Vacuum is expressed in mbar or Pa pressure units.

A1.3.2 *Target or Test Vacuum (Vac)*—The vacuum level in the test chamber is referred to as either Target or Test Vacuum. Target Vacuum is the vacuum level the instrument is programmed to achieve during the first phase of the test cycle. Once Target Vacuum is achieved, the vacuum source is automatically isolated from the test chamber and the test cycle proceeds. During the remainder of the test cycle the actual vacuum level in the test chamber is referred to as Test Vacuum. Target or Test Vacuum is expressed in mbar or Pa pressure units.

A1.3.3 *Reference Vacuum (VRef)*—Reference Vacuum is the minimum vacuum level that must be maintained in the test chamber throughout the length of the cycle. VRef is a vacuum level somewhat less than Target or Test Vacuum (Vac), and is expressed in mbar or Pa pressure units.

A1.3.4 *Reference Fill Time (TfillRef)*—The allotted time for achieving Target Vacuum is called the Reference Fill Time. The actual recorded time required is Fill Time (Tfill). Both TfillRef and Tfill are expressed in time units of seconds.

A1.3.5 *Equalization Time (Tequal)*—Equalization Time is the time in seconds allowed for any "settling" of pressure fluctuations in the test chamber after initial vacuum has been drawn.

A1.3.6 *Test Time (Ttest)*—Immediately after Tequal, the Test Time period of the test cycle takes place. During Ttest the Vacuum Decay Rate (dP/dt) is monitored (see A1.3.7), as well as the Test Vacuum (Vac) (see A1.3.2). Ttest is expressed in time units of seconds.

A1.3.7 *Reference Vacuum Decay Rate (dP/dtRef)*—During Ttest, the change in vacuum as a function of time within the test chamber is monitored (Vacuum Decay Rate [dP/dt]). The maximum allowable vacuum loss is referred to as Reference Vacuum Decay Rate (dP/dt Ref), and is expressed in units of mbar per second, or Pa per second.

A1.4 Test packages are identified as Rejects (FAIL) when any one of the following occurs.

A1.4.1 Target Vacuum (Vac) is not achieved within the allotted Reference Fill Time (TfillRef).

A1.4.2 Test Vacuum (Vac) drops below the Reference Vacuum (VRef) during the Equalization Time (Tequal) or Test Time (Ttest).

A1.4.3 Test chamber Vacuum Decay Rate (dP/dt) exceeds the Reference Vacuum Decay Rate (dP/dtRef) during the Test Time (Ttest).

A1.5 Test packages are identified as Accept (PASS) when all of the following criteria are met.

A1.5.1 Target Vacuum (Vac) is achieved within the Reference Fill Time (TfillRef).

A1.5.2 Test chamber vacuum (Test Vacuum [Vac]) remains at or above the Reference Vacuum (VRef) throughout the Equalization Time (Tequal) and Test Time (Ttest).

A1.5.3 Test chamber Vacuum Decay Rate (dP/dt) remains less than or equal to the Reference Vacuum Decay Rate (dP/dtRef) throughout the Test Time (Ttest).

A2. ESTABLISHING CRITICAL TEST PARAMETERS AND VERIFYING TEST SENSITIVITY

A2.1 Establishing critical test parameters of cycle times (TfillRef, Tequal, and Ttest), pressures (VRes, Vac, VRef), and vacuum decay rate (dP/dtRef) requires some expertise and experience with the packages in question. The user is advised to check the instrument's operating manual for more detailed instructions. Two approaches that may be used to establish leak test critical parameters are described below:

A2.1.1 *Manual Approach*:

A2.1.1.1 A population of control, non-leaking packages is exposed to various vacuum levels for lengthy Tfill periods of 1 or more seconds to determine a Target or Test Vacuum (Vac) that is great enough to allow for significant package leakage without causing package seal failure, and to determine the typical time period necessary for achieving this level of vacuum (Tfill). Once the Target Vacuum (Vac) has been selected, a VRes value about 10 % higher than the Target Vacuum is selected. Time observations will allow a selection of TfillRef (Reference Fill Time) and Tequal (Equalization Time). Both TfillRef and Tequal are typically less than 1 second.

A2.1.1.2 Other critical parameters are selected by observing the test chamber pressure change over a lengthy test cycle of a few seconds for the control package population. These baseline vacuum decay data are used to select the remaining critical parameters of dP/dtRef (Reference Vacuum Decay Rate), VRef (Reference Vacuum), and Ttest (Test Time).

A2.1.2 *Automatic Approach*:

A2.1.2.1 Some instruments may be equipped with an automatic test cycle selection function. When operated in this mode, a population of control, non-leaking packages are tested according to a pre-set cycle. The instrument then automatically selects optimum test parameter conditions that reflect the performance of these control packages.

A2.2 *Test Qualification*:

A2.2.1 After critical test parameters have been identified, it is important to verify the ability of the test to successfully identify defective packages. Successful defect detection is a function of the critical test parameters, as well as the configuration of the test chamber. Successful tests are also related to the location and type of package defects present. For example, leaks can become clogged with product or they may be pinched off or masked when closed inside the test chamber. (Refer to Sections 4 and 6 for further information.)

A2.3 *Test Sensitivity Verification*:

A2.3.1 Test sensitivity is verified by introducing a known volumetric flow rate of air into the test system containing a

non-leaking package during the test cycle. The sensitivity is defined as the minimum airflow rate that will trigger a Reject or FAIL result. Sensitivity is specific for a given package type/test chamber combination for the critical test parameters selected. This approach was utilized in the study described in 12.2.

A2.3.2 Alternatively, it may be desirable to define a test's sensitivity according to the nature and size of the package defects reliably detected. The reliability of this approach depends on the quality of the defective samples; precisely made defects are often difficult to prepare and maintain. Therefore, the user is advised to use defective packages for test qualification (see A2.2) and to establish test sensitivity using a calibrated volumetric airflow meter.