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

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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.

1.2 *Leaks Detected*—This test method detects package leaks by measuring the rise in pressure (vacuum loss) in an enclosed evacuated test chamber containing the test package. Vacuum loss results from leakage of test package headspace gases and/or volatilization of test package liquid contents located in or near the leak. When testing for leaks that may be partially or completely plugged with the package's liquid contents, the test chamber is evacuated to a pressure below the liquid's vaporization pressure. All methods require a test chamber to contain the test package and a leak detection system designed with one or more pressure transducers. Test method sensitivities cited below were determined using specific product-package systems selected for the precision and bias studies summarized in Table 1. Table 1 also lists other examples of relevant product-package systems that can be tested for leakage by vacuum decay.

1.2.1 *Trays or Cups (Non-lidded) (Air Leakage)*—Hole or crack defects in the wall of the tray/cup of at least 50 µm in diameter can be detected. Nonlidded trays were tested at a Target Vacuum of –4·E4 Pa (–400 mbar).

1.2.2 *Trays Sealed with Porous Barrier Lidding Material (Headspace Gas Leakage)*—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·E4 Pa (–400 mbar). The sensitivity of the test for porous lidded packages is approximately E-2 $Pa{\cdot}m^3{\cdot}s^{-1}$ using a calibrated volumetric airflow meter.

1.2.3 *Rigid, Nonporous Packages (Headspace Gas Leakage)* —Hole defects of at least 5 µm in diameter can be detected. Plastic bottles with screw caps were tested at a target vacuum of –5·E4 Pa (–500 mbar). Using a calibrated volumetric airflow meter, the sensitivity of the test is approximately E-3.4 $Pa{\cdot}m^3{\cdot}s^{-1}$. Air-filled glass syringes were tested at a target vacuum of –7.5·E4 Pa (+250 mbar absolute) and again at a target vacuum of about +1 mbar absolute. The sensitivity of both tests is approximately E-4.1 $Pa{\cdot}m^3{\cdot}s^{-1}$ using a calibrated volumetric airflow meter.

1.2.4 *Rigid, Nonporous Packages (Liquid Leakage)*—Hole defects of at least 5 µm in diameter can be detected. This detection limit was verified using a population of water-filled glass syringes tested at a target vacuum of about +1 mbar absolute.

1.2.5 *Flexible, Nonporous Packages (Gas or Liquid Leakage)* —Such packages may also be tested by the vacuum decay method. 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*—Test results are qualitative (Accept/Reject). Acceptance criteria are established by comparing quantitative baseline vacuum decay measurements obtained from control, non-leaking packages to measurements obtained using leaking packages, and to measurements obtained with the introduction of simulated leaks using a calibrated gas flow meter.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

[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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TABLE 1 Summary of Vacuum Decay Leak Tests Applications for Various Product-Packages Systems

Package Examples[A]	Package Content Examples	ASTM P&B Data Tables	Target Vacuum[B]
GAS LEAK TEST PACKAGE APPLICATIONS AND PRECISION & BIAS STUDIES			
Porous barrier lidded trays[C]	Empty Solids (tablets, capsules, powders, devices)	3, 4, 5	–400 mbar
Nonlidded trays[C] or cups	Empty	2	–400 mbar
Plastic screw capped bottles[C]	Solids (tablets, capsules, powders) Liquids (with significant gas headspace volume)	6	–500 mbar
Glass syringes[C]	Solids (lyophilized powders)	7, 8	+250 mbar
ADDITIONAL GAS LEAK TEST PACKAGE APPLICATIONS[A]			
Lidded (nonporous) trays or cups containing solid materials (for example, powders, tablets, capsules, devices)			
Glass or plastic vials closed with elastomeric closures containing solid materials (for example, powders)			
Glass or plastic vials closed with elastomeric closures, containing liquid materials, but with significant gas headspace volume			
Flexible packages (for example pouches or bags) containing solid materials (for example, powders, devices)			
LIQUID LEAK TEST (with or without gas headspace) PACKAGE APPLICATIONS AND PRECISION & BIAS STUDIES			
Glass syringes[C]	Liquids	9, 10	+1 mbar
ADDITIONAL LIQUID LEAK TEST PACKAGE APPLICATIONS[A]			
Ophthalmic dropper tip bottles containing liquid materials			
Glass or plastic ampoules containing liquid materials			
Glass or plastic vials with elastomeric closures containing liquid materials			
Lidded (nonporous trays or cups) containing liquid materials			
Flexible packages such as pouches or bags containing liquid materials			

[A] Examples of package types relevant to the specified leak test method are listed. The list is not intended to be all inclusive.
[B] Target vacuum expressed as a negative mbar reading (e.g., –400 mbar) refers to the measured test chamber pressure (vacuum) relative to atmospheric pressure. Target vacuum expressed as a positive mbar reading (e.g., +1 mbar) refers to the absolute pressure reading in the test chamber.
[C] Packages used for the referenced ASTM Precision and Bias (P&B) studies.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D996 Terminology of Packaging and Distribution Environments

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F17 Terminology Relating to Flexible Barrier Packaging

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

3. Terminology

3.1 *Definitions*—For definitions used in this test method, see Terminologies D996, F17, and F1327.

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 *gas leaks, n*—leak paths that allow the flow of gas from the test package.

3.2.5 *liquid leaks, n*—leak paths partially or fully filled with liquid.

3.2.6 *rigid, nonporous packages, n*—packages that do not significantly deflect under vacuum and are constructed of solid, nonporous materials. Examples include plastic bottles with screw-thread or snap-on closures, glass or plastic vials with elastomeric closures, and glass or plastic syringes.

3.2.7 *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.

3.2.8 *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.

3.2.9 *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

[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.

vacuum source and a pressure transducer (absolute or gauge) alone or in combination with a second differential pressure 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. Vacuum decay can also result from the volatilization of packaged liquid that partially or fully occludes the leak path. In this case, vacuum decay will only occur if the chamber test pressure is lowered below the liquid's vaporization pressure.

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 test is a function of test package design, transducer(s)'sensitivity, test chamber design, test system design, and critical test parameters of time and pressure. The test system and leak test parameters selected for any given product-package system must be based on the package's contents (liquid or solid with significant or little gas headspace), and the nature of the package (flexible or rigid, porous or nonporous). Instruments with more sensitive pressure transducers and with minimal void volumes within the test chamber and the test system have the potential to detect the smallest leaks. Lengthening test time enables smaller gaseous leaks to be detected. Minimizing pressure variation background noise can also improve test sensitivity. For porous barrier lidded packages, masking techniques will minimize background noise. For flexible or semi-rigid packages, restricting package expansion via properly designed test chambers lessens noise. Background noise may also occur upon release of residual gases or vapors trapped in the test system or between test package components. Such noise can be differentiated from actual leakage by lengthening the time to reach initial vacuum or lengthening equalization time.

NOTE 1—Further information on the "Leak Test Theory" may be found in Annex A1. Examples of test methods and test equipment used to generate the precision and bias data in Section 12 are summarized in Table 1.

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, individual test operation may be semi-automatic, automatic, or manual. The test method permits 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 package porous barrier surface during the test to prevent 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 resulting from gas or vapor egress from a package challenged with vacuum.

5.3 This test is a useful research tool for optimizing package sealing parameters and for comparatively evaluating 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*—The vacuum decay leak apparatus includes a test chamber connected to a vacuum decay test system and a volumetric airflow meter.

6.2 *Test Chamber*—The test chamber has 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. Figs. 2 and 3 illustrate test chambers designed for testing rigid, nonporous packages. In the latter two cases, there is no flexible bladder.

6.2.1 *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.

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

6.3 *Vacuum Decay Test System*—The vacuum decay test system includes a vacuum source for establishing the required vacuum within the chamber at the beginning of the test cycle, and a pressure transducer (absolute or gauge), alone or in combination with a second differential pressure transducer, for monitoring the vacuum level as well as the pressure change as a function of time during the test cycle. Test systems intended for higher target vacuums, such as +1 mbar or less, should be designed for greater target pressure measurement accuracy,

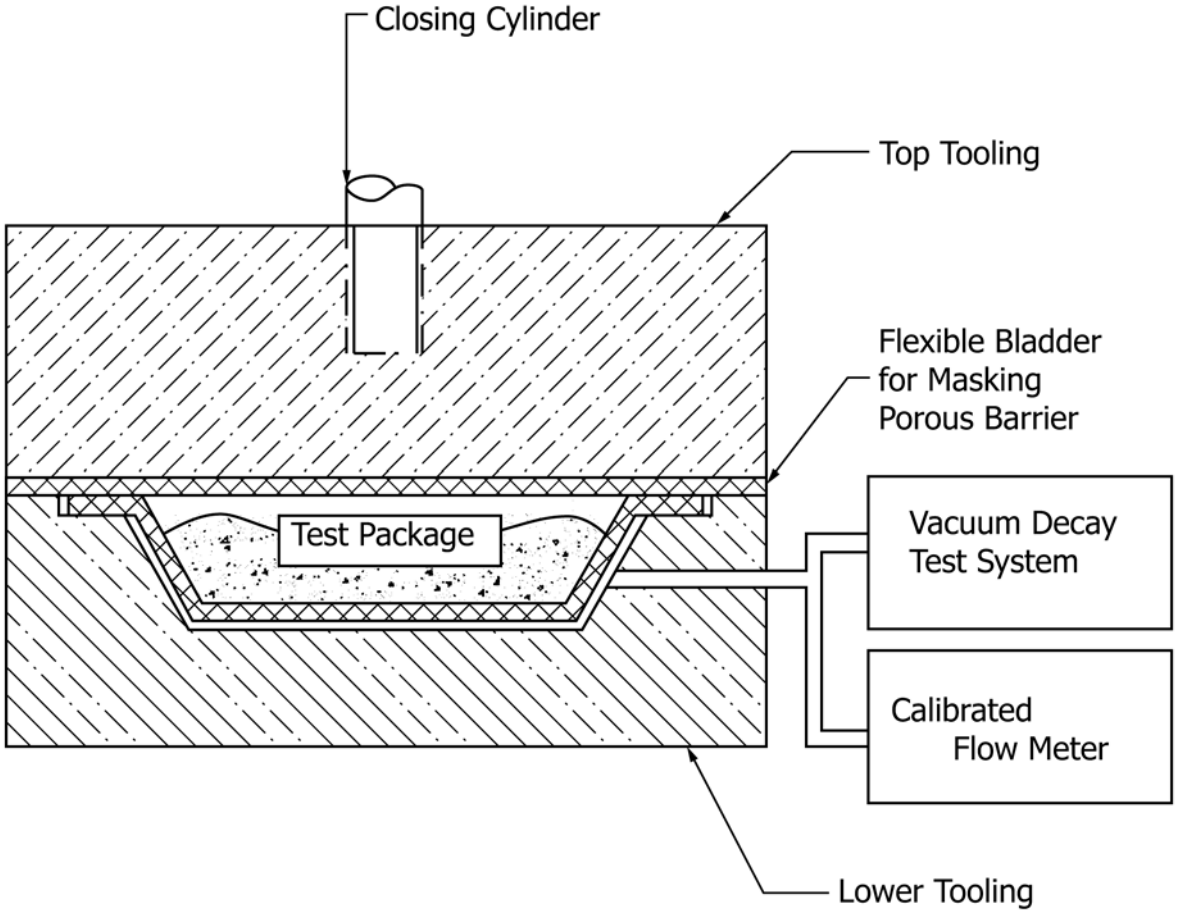

FIG. 1 Schematic of Fixture and Porous Barrier Lidded Test Package

with minimal system leakage and outgassing that may affect test measurement signal to noise ratio.

NOTE 2—Different leak test instruments may utilize different pressure transducer types and combinations, and vacuum pumps based on the package types tested (for example, rigid versus nonrigid, porous versus nonporous) and the vacuum level that is required to perform the test.

6.3.1 *Absolute versus Gauge Transducer*— All instruments includes a single 1000 Torr transducer for monitoring test pressure throughout the test cycle. An absolute transducer is preferred over a gauge transducer when precise, true pressure readings are required (that is, not subject to atmospheric pressure changes from weather or altitude). Such is the case when performing high vacuum liquid leak tests.

6.3.2 *Differential Transducer*—A second differential pressure transducer may be employed for measuring the smallest detectable leaks in rigid or semi-rigid nonporous packages.

6.3.3 *Vacuum Source*—A vacuum pump is selected based on the target vacuum level that must be achieved within the allotted time frame given the test system airspace.

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 at variable rates. It is recommended that an airflow meter be used to verify the leak test's sensitivity.

NOTE 3—Refer to Annex A2 for further information about volumetric airflow meter use 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. For those instruments that rely on an internal, air-driven vacuum pump, the utilities required for instrument operation include electrical power and a dry, non-lubricated compressed air supply, according to manufacturer's specifications. For those instruments that rely on an external vacuum pump, the utilities required for instrument operation include electrical power according to manufacturer's specifications for both the instrument and the vacuum pump.

9. Calibration and Standardization

9.1 Before test measurements are made, the apparatus must be calibrated. The pressure transducers, any applicable vacuum

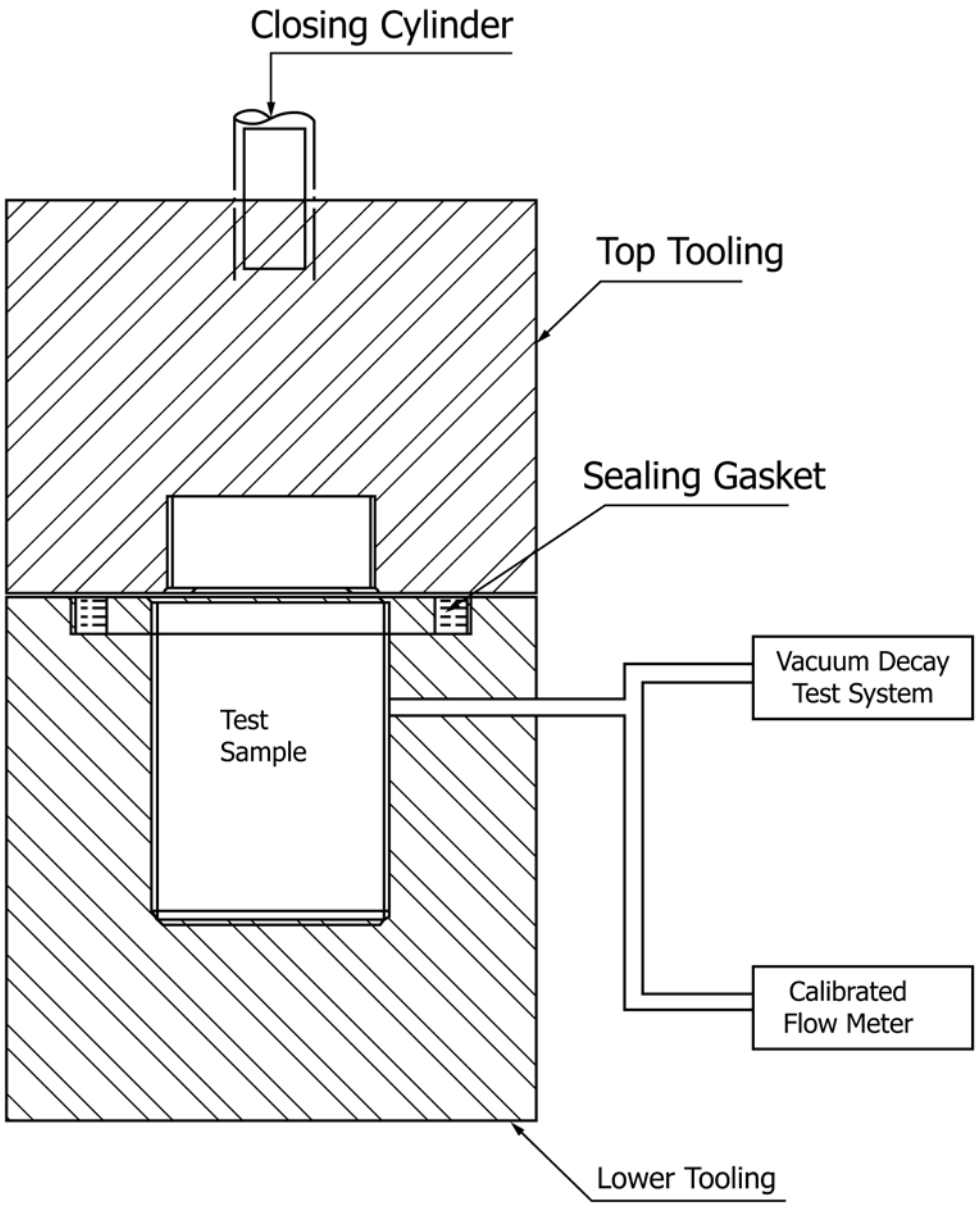

FIG. 2 Schematic of Fixture and Rigid, Nonporous Test Package

source pressure gauges, and the adjustable volumetric airflow meter must all be calibrated according to the manufacturer's recommended procedures and maintenance schedule.

9.2 Leak tests should be performed on the instrument test system to verify a steady baseline leak rate. The test parameters for start-up system qualification tests are typically recommended by the instrument manufacturer.

9.3 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. A few control non-leaking packages or a no-leak package mock-up must be used to select critical test parameter settings.

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

9.4 A larger sample population of control non-leaking packages must be used for 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 5—Refer to Annex A2 for information on critical test parameter selection.

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

NOTE 6—Refer to Annex A2 for information about test sensitivity verification procedures.

9.6 Qualify the ability of the optimized test to reliably differentiate between known non-leaking and defective packages.

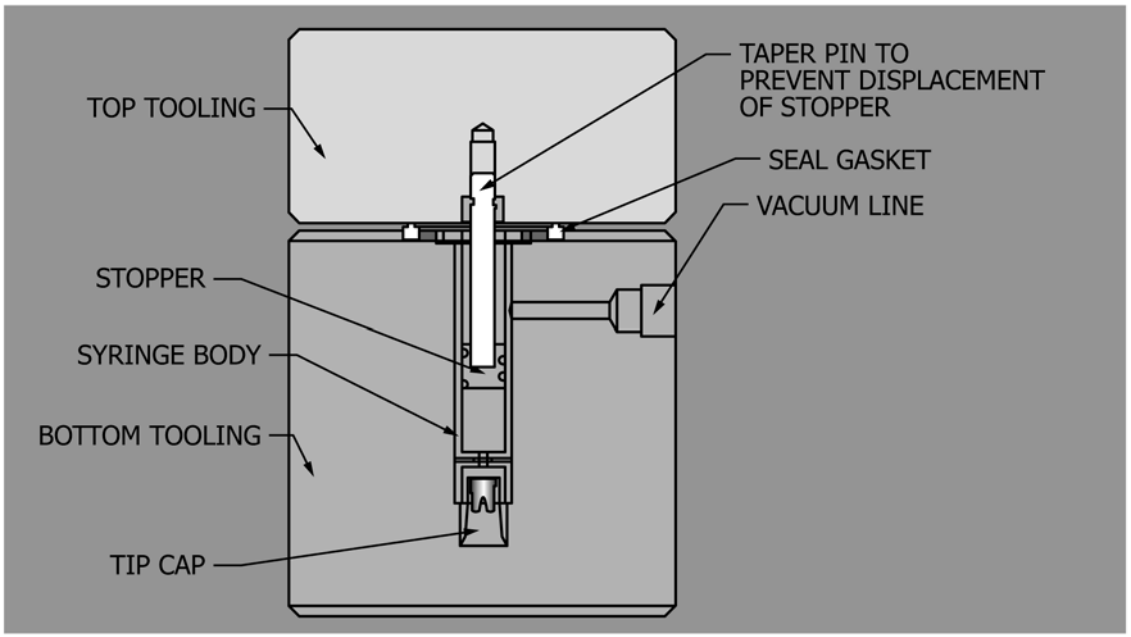

FIG. 3 Schematic of Test Chamber and Rigid, Nonporous Test Package

9.7 Test system baseline qualification (see 9.2) 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.

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 For those test methods that require a Pre-Test Vacuum sequence prior to each test sample leak test, close the empty test chamber and perform the required timed vacuum sequence.

10.5 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 7—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.

10.6 Start the test.

10.7 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.

10.7.1 If suspect fail results occur, verify the test chamber and system functionality according to the leak test instrument manufacturer's instructions prior to proceeding.

10.7.2 If a failed test package contains product that may have contaminated the test chamber or system during the leak test, perform steps to eliminate the contaminant from the test chamber or system according to the leak test instrument manufacturer's instructions prior to proceeding.

10.8 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 Pre-Test Vacuum expressed in seconds.

11.1.2 Reserve Vacuum expressed in mbar or Pa, in either positive absolute pressure units or negative pressure units (vacuum) relative to atmospheric pressure.

11.1.3 Target Vacuum expressed in mbar or Pa, in either positive absolute pressure units or negative pressure units (vacuum) relative to atmospheric pressure.

11.1.4 Reference Vacuum expressed in mbar or Pa, in either positive absolute pressure units or negative pressure units (vacuum) relative to atmospheric pressure.

11.1.5 Reference Fill Time expressed in seconds.

11.1.6 Equalization Time expressed in seconds.

11.1.7 Test Time expressed in seconds.

11.1.8 Reference Vacuum Decay Accept/Reject Limit expressed in Pa/s or Pa, in either positive absolute pressure units to describe allowable pressure rise, or negative pressure units (vacuum) to describe allowable vacuum loss.

11.1.9 Accept/Reject Test Results.

NOTE 8—Refer to Annex A1 for definitions of critical test parameters. The nomenclature used to describe critical test parameters may vary with the equipment manufacturer, but the essential definitions remain unchanged.

TABLE 2 Gas Leak Detection Results—Nonlidded Tray

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 %).

12. Precision and Bias

12.1 *Precision:*

NOTE 9—Refer to Table 1 for a summary of the various test equipment, test methods and packages used to generate the precision and bias data presented.

NOTE 10—All test results are expressed in qualitative terms (accept/reject). Precision and bias studies indicate the percentage of packages meeting the test criterion.

NOTE 11—The vacuum decay instruments used in this round robin were manufactured by Packaging Technologies and Inspection. All available apparatus may not be suitable for this application. Apparatus considered for use in this application shall be checked for suitability in accordance with the requirements in Section 6.

12.1.1 *Gas Leak Detection:*

12.1.1.1 *Nonlidded and Porous Barrier Lidded Trays*—An interlaboratory study was run in accordance with Practice E691 using a single pressure transducer (gauge) vacuum decay instrument.[4] 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 by means of various adhesive systems. The same test samples were tested at each laboratory.[5] 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. Another single laboratory study was run testing the same vacuum decay instrument's ability to detect air flow leaks introduced into in test chambers containing packages with various porous barrier lidding material types.

(1) Nonlidded Trays—The test method is able to identify defective trays with holes ≥50 µm, when using a Target Vacuum (Vac) of –4·E4 Pa (–400 mbar). As summarized in Table 2, 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. These two trays were eliminated from the precision statement.

(2) Porous Barrier Lidded Trays—The test method is able to identify defective packages sealed with porous barrier lidding material, 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·E4 Pa (–400 mbar). As per the results outlined in Table 3, 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.

(3) Porous Barrier Lidded Trays with Various Adhesive Bonding Systems— The test method is able to reliably identify packages with less than optimum seal bonding for dot matrix adhesive systems, and severely incomplete bonds made with continuous adhesive systems at a Target Vacuum of –4·E4 Pa (–400 mbar). Table 4 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.

(4) Trays with Various Porous Barrier Lidding Materials—The test method can be used to test packages sealed with various porous barrier lidding material types, and tests are similar in sensitivity (approximately E-2 $Pa \cdot m^3 \cdot s^{-1}$ at a Target Vacuum of –4·E4 Pa [–400 mbar]). Table 5 summarizes a single laboratory study run using a single pressure transducer (gauge) vacuum decay instrument[4] to verify the test method's ability to evaluate semi-rigid thermoformed tray packages sealed with various porous barrier lidding material types, and to obtain an estimate of the tests' sensitivity.[5] Critical test parameters were identified for each package population. Each

[4] Model Pti VeriPac 225 by Packaging Technologies and Inspection, 145 Main Street, Tuckahoe, NY 10707. See Note 11.

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

TABLE 3 Gas Leak Detection Results—Trays with Porous Barrier Lidding

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 4 Gas Leak Test Results—Trays with Porous Barrier Lidding Seal Bonding Defect

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.

TABLE 5 Gas Leak Test 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 (16.5 × 13 × 2.5)	A	C	0	0	15	0	15	100
			26	4·E-2	4	1	3	25
			29 to 52	(5 to 9) E-2	16	16	0	100
536 cm^3 (16.5 × 13 × 2.5)	A	D	0	0	15	0	15	100
			17	3·E-2	5	2	3	40
			19 to 35	(3 to 6) E-2	15	15	0	100
536 cm^3 (16.5 × 13 × 2.5)	B	E	0	0	4	0	4	100
			13 to 21	(2 to 3) E-2	6	3	3	50
			22 to 34	(4 to 6) E-2	6	6	0	100

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

test's sensitivity was determined by introducing air via a calibrated volumetric airflow meter into the instrument test chamber containing the test package. The test's sensitivity was defined as the leak rate that first triggered FAIL test results.

12.1.1.2 *Rigid, Nonporous Packages*—Two studies, one evaluating rigid HDPE bottles with induction seals and screw-caps, and another evaluating glass syringes with staked needles, were performed utilizing two differently designed instruments to detect gas leaks in rigid, nonporous packages. These studies are described below.

(1) HDPE Bottles—The test method is able 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 of –5·E4 Pa (–500 mbar). No control packages were falsely rejected. Test method is able to detect a calibrated gas flow rate of between 0.25 and 0.27 ccm (equivalent to a volumetric flow rate at target vacuum of E–3.4 to E–3.3 $Pa \cdot m^3 \cdot s^{-1}$). Table 6 summarizes a single laboratory study run using three identical vacuum decay instruments, designed with a pressure transducer (gauge) combined with a differential pressure transducer,[6] to verify the test method's ability to evaluate rigid, nonporous packages, and to obtain an estimate of the tests' sensitivity.[7] The packages tested included

[6] Model Pti VeriPac 325 by Packaging Technologies and Inspection, 145 Main Street, Tuckahoe, NY 10707. See Note 11.

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

TABLE 6 Gas 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)	Success Rate (% accurate tests)
30-mL, Induction Seal, Screw-thread bNon-Child-Resistant Closure	No defect	35	315	0	315	100
	<5 µm hole	3	27	25	2	93
	5 µm hole	3	27	27	0	100
	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, Screw-thread Push and Turn Child-Resistant Closure	No defect	35	315	0	315	100
	<5 µm hole	3	27	26	1	96
	5 µm hole	3	27	27	0	100
	10 µm hole	3	27	27	0	100
	25 µm hole	3	27	27	0	100
	50 µm hole	3	27	27	0	100

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 introducing a single laser-drilled hole in the induction seal face (including holes <5, 5, 10, 25, and 50 µm in diameter). Holes rated as less than 5 µm could not be reliably sized microscopically. After defects were created, caps were torqued onto the bottles. Three replicate tests were performed on each test sample using each test instrument. The same Critical Test Parameters were utilized for all three test units, for all test sample populations. Test method sensitivity was determined by introducing a gas flow into the test chamber using a calibrated volumetric flow-meter and determining the flow rate that first triggered FAIL test results for all control, pilot samples tested.

(2) Glass Syringes—The test method is able to identify defective packages with holes at least 5.0 µm in diameter when tested at +2.5E–4 Pa (+250 mbar absolute). No control packages were falsely rejected. One false negative test result occurred when testing the package with the smallest size defect (4.7 µm). The test method is able to identify a volumetric gas flow rate of 0.05 ccm (equivalent to a volumetric flow rate of E–4.1 $Pa{\cdot}m^3{\cdot}s^{-1}$ at target vacuum).Table 7 summarizes an interlaboratory study performed at three participating laboratories, using three vacuum decay instruments each utilizing a pressure transducer (absolute) in combination with a differential pressure transducer[8] to verify the test method's feasibility in evaluating rigid, nonporous packages, and to estimate how well such artificially induced leaks in syringes can be detected.[9] The packages tested included glass syringes, 1-mL capacity, with staked needle. Defective packages were made by the introduction of a single laser-drilled hole in the glass syringe barrel (hole sizes were approximately 5, 10, and 15 µm in nominal diameter). Both control (no defect) and defective syringes were tested empty (air-filled) simulated packages that contain dry product such as a lyophilized powder, or packages with gas headspace at the leak site. Each test site performed three replicate tests on each control, no defect test sample using each test instrument. A separate group of defective, holed syringes was tested in triplicate at each test site to minimize the risk of hole clogging. The same Critical Test Parameters were utilized for all three test units, for all test sample populations. Test method sensitivity was determined using a calibrated volumetric airflow meter to introduce an air flow leak into the instrument test chamber containing a series of control test packages and determining the leak rate that first triggered FAIL test results. Results are summarized in Table 8.

12.1.1.3 *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.

12.1.2 *Liquid Leak Detection:*

12.1.2.1 *Rigid, Nonporous Packages*—One study evaluating water-filled glass syringes with staked needles was performed to detect leaks in liquid-filled rigid, nonporous packages. This study is described below.

(1) Glass Syringes—The test method is able to identify defective packages with holes at least 5.0 µm in diameter when tested at about +1 mbar absolute, regardless of the presence of liquid in the leak path. Liquid presence improved leak test method sensitivity by causing a greater vacuum decay. All negative control packages results were accurate with no false rejects. The test method is able to detect a calibrated gas flow rate of 0.05 ccm (equivalent to volumetric flow rate of E–4.1 $Pa{\cdot}m^3{\cdot}s^{-1}$ at target vacuum). Table 9 summarizes an interlaboratory study performed at three participating laboratories, using three vacuum decay instruments each utilizing a pressure transducer (absolute) in combination with a differential pressure transducer[8] to verify the test method's feasibility in evaluating rigid, nonporous packages, and to obtain an estimate the test's ability to detect artificially induced leaks in syringes. The packages tested included glass syringes, 1-mL capacity, with staked needle. Control syringes, without defect, were filled with water. Defective packages were made by introducing a single laser-drilled hole in the glass syringe barrel (hole sizes were approximately 5, 10, and 15 µm in nominal diameter). The defective packages were tested both empty (air-filled) and water-filled. This was done to demonstrate the leak test method's ability to detect leaks located either in the gas headspace region or at the liquid product level. Three replicate tests were performed on each control, no defect test

[8] Model Pti VeriPac 325–LV by Packaging Technologies and Inspection, 145 Main Street, Tuckahoe, NY 10707. See Note 11.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1027.

TABLE 7 Gas Leak Test Results—Rigid, Nonporous Glass Syringes, 1 mL, Air-filled

Defect Description	Number of Package Units Tested	Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	Success Rate (% accurate tests)
No defect	15	135	0	135	100
5 µm hole	15	45	44	1[A]	98
10 µm hole	15	45	45	0	100
15 µm hole	15	45	45	0	100

[A] The false negative result test sample hole size was 4.7 µm.

TABLE 8 Gas Leak Test Results—Control, No Defect Glass Syringes, 1 mL Air-filled

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)
0	0	135	0	135	100
0.050	E-4.1	45	43	2	96
0.100	E-3.8	45	45	0	100

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

TABLE 9 Liquid Leak Test Results—Rigid, Nonporous-Glass Syringes, 1 mL Air- and Water-Filled

Package	Defect Description	Number of Package Units Tested	Number of Replicate Tests	Number FAILED (Leaks detected)	Number PASSED (No leaks detected)	Success Rate (% accurate tests)
1 mL Glass Syringe, Staked Needle, Water-Filled Defects	No defect (water-filled)	15	134	0	134	100
	5 µm hole	15	45	45	0	100
	10 µm hole	15	45	45	0	100
	15 µm hole	15	45	45	0	100
1 mL Glass Syringe, Staked Needle, Air-filled Defects	No defect (water-filled)	15	135	0	135	100
	5 µm hole	15	45	45	0	100
	10 µm hole	15	45	45	0	100
	15 µm hole	15	45	45	0	100

NOTE 1—All no-defect syringes were water-filled. Air-filled defects simulate leaks found in the gas headspace region of packages filled with liquid.

sample using each test instrument. A separate group of defective, holed syringes was tested in triplicate at each test site to minimize the risk of hole clogging. The same Critical Test Parameters were utilized for all three test units, for all test sample populations. Test method sensitivity was determined using a calibrated volumetric airflow meter to introduce an air flow leak into the instrument test chamber containing a series of control test packages and determining the leak rate that first triggered FAIL test results. Results are summarized in Table 10.

12.2 *Bias*—No bias calculations are possible for studies described in 12.1 as there are no accepted reference numerical values to compare to test results.

13. Keywords

13.1 absolute pressure transducer; barrier performance; block; criteria parameters; differential pressure transducer; flexible packaging; food packages; gauge pressure transducer; hole leaks; holes; leaks; liquid 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

TABLE 10 Liquid Leak Test Results—Control, No Defect Glass Syringes, 1 mL Water-Filled

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)
0	0	135	0	135	100
0.050	E-4.1	45	45	0	100
0.100	E-3.8	45	45	0	100

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

ANNEXES

(Mandatory Information)

A1. VACUUM DECAY LEAK TEST THEORY

A1.1 A vacuum decay leak test procedure is performed by exposing a test package to an external vacuum. The differential pressure applied to the package draws out gases through leakage pathways. If the package contents include liquid, a vacuum level below the liquid's vaporization pressure will also volatilize liquid in or near leakage pathways. The rise in test chamber pressure during a test cycle, as monitored by one or more pressure transducers, is the result of internal package headspace gases and/or volatilized liquids 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 or vapors in the test chamber or test system lines. Background noise may be minimized by test chamber design modifications, adjustments to pressure or time parameters, or by exposing the test chamber and system to vacuum for a predetermined time sequence prior to loading the test sample into the test chamber.

A1.2 Packages that include a porous barrier lidding material can be tested for gas leaks 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 package 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 in the test chamber is 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 as well as the leak test acceptance criteria are described below. Fig. A1.1 illustrates a typical test cycle, with the various leak test failure modes indicated.

NOTE A1.1—Critical test parameter nomenclature used in the following text may vary with leak test equipment manufacturer, although the definitions remain the same.

A1.3.1 *Reserve Vacuum*— Reserve Vacuum is expressed in mbar or Pa pressure units. Some equipment describe Reserve Vacuum in absolute pressure terms, while others express Reserve Vacuum as vacuum (negative pressure) relative to atmospheric pressure. In vacuum terms, Reserve Vacuum should be somewhat greater than Target Vacuum. In absolute pressure terms, the Reserve Vacuum pressure should be somewhat less than Target Vacuum pressure.

A1.3.2 *Pre-Test Vacuum Flush*—A Pre-Test Vacuum Flush is the period of time (expressed in seconds) when the empty test chamber and test system are held under Reserve Vacuum conditions just prior to initiating a test sample leak test. Pre-Test Vacuum Flush is not required to perform a leak test, but may be used to maximize leak test method sensitivity by minimizing background noise.

A1.3.3 *Target 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. Target Vacuum is expressed in mbar or Pa pressure units.

A1.3.4 *Test Vacuum*— Test Vacuum is the measured test chamber vacuum level throughout the test cycle. Test Vacuum is expressed in mbar or Pa pressure units. Some equipment describe Test Vacuum as vacuum (negative pressure) relative to atmospheric pressure, while other equipment describe Test Vacuum in absolute pressure terms. Both are illustrated in Fig. A1.1.

A1.3.5 *Fill Time and Reference Fill Time*—The Reference Fill Time is the allotted time for achieving Target Vacuum. The actual time necessary to reach Target Vacuum is the Fill Time. Both the Fill Time and the Reference Fill Time are expressed

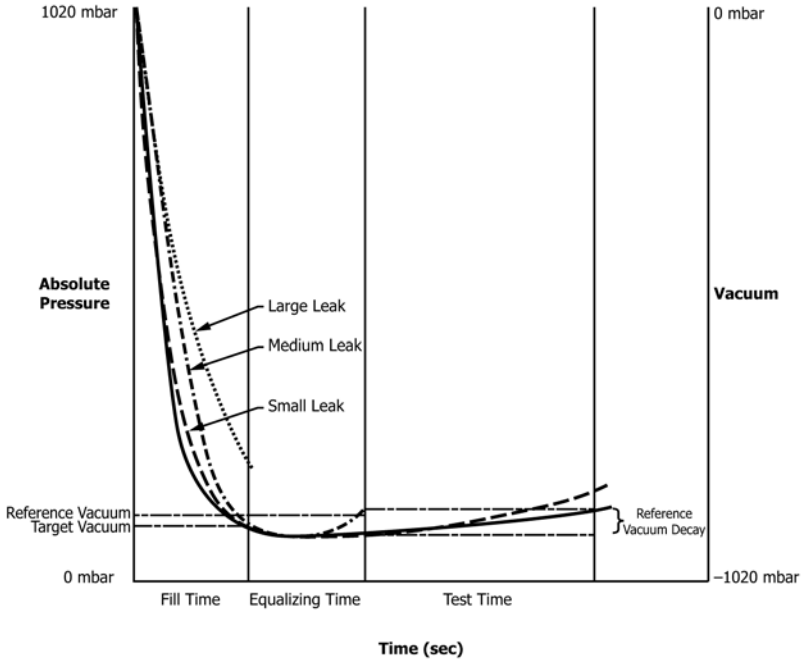

FIG. A1.1 Vacuum Decay Leak Rate Profiles

in time units of seconds. If the test cycle is programmed to monitor the rise in vacuum (that is, the drop in absolute pressure) in stages, then multiple Reference Vacuum settings and Reference Fill Time specifications apply.

A1.3.6 *Equalization Time*—Equalization Time immediately follows Fill Time. Equalization time (expressed in time units of seconds) allows for any "settling" of pressure fluctuations in the test chamber and allows for trapped gases around the test package to escape (for example from around cap screw threads). Equalization time is typically a few seconds. However, Equalization Time may be very short (<1 s) when it is necessary to detect a rapid pressure rise (that is, loss of vacuum) from the volatilization of liquid in leak spaces.

A1.3.7 *Test Time*—Test Time (expressed in time units of seconds) takes place immediately after Equalization Time. During Test Time the Test Vacuum is continually monitored for evidence of package leakage. The same pressure transducer is used to measure Test Vacuum during Fill Time, Equalization Time and Test Time. A second differential pressure transducer with greater sensitivity may also be used during Test Time for detecting pressure changes from the smallest leaks.

A1.3.8 *Reference Vacuum*—Reference Vacuum defines the vacuum level that must be maintained in the test chamber after Target Vacuum is achieved and throughout Equalization Time and Test Time. Reference Vacuum is a vacuum level somewhat less than Target or Test Vacuum and is expressed in mbar or Pa pressure units. In absolute pressure terms, Reference Vacuum pressure is somewhat greater than Target or Test Vacuum absolute pressure.

A1.3.9 *Reference Vacuum Decay*—Reference Vacuum Decay defines the maximum allowable drop in vacuum (that is, rise in absolute pressure) during Test Time. Reference Vacuum Decay may be expressed in units of pressure (Pa) or in units of pressure change (Pa/s).

A1.4 Test packages are identified as Rejects (FAIL) when any one of the following occurs. Failure modes are illustrated in Fig. A1.1.

A1.4.1 Target Vacuum is not achieved within the allotted Reference Fill Time.

A1.4.2 Test Vacuum drops below the Reference Vacuum (that is, Test Vacuum absolute pressure rises above the Reference Vacuum absolute pressure) during Equalization Time or Test Time.

A1.4.3 Test chamber Vacuum Decay (or pressure rise) exceeds the Reference Vacuum Decay (or allowable pressure rise) during the Test Time.

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

A1.5.1 Target Vacuum is achieved within the Reference Fill Time.

A1.5.2 Test chamber vacuum meets or exceeds the Reference Vacuum (that is, Test chamber absolute pressure remains equal to or lower than Reference Vacuum absolute pressure) throughout the Equalization Time and Test Time.

A1.5.3 Test chamber Vacuum Decay remains less than or equal to the Reference Vacuum Decay (that is, Test chamber pressure rise remains equal to or lower than Reference Vacuum pressure rise) throughout the Test Time.

A2. ESTABLISHING CRITICAL TEST PARAMETERS AND VERIFYING TEST SENSITIVITY

A2.1 Establishing critical test parameters of cycle times (Pre-Test Vacuum Flush, Fill Time, Equalization Time, and Test Time), pressures (Reserve Vacuum, Target Vacuum, and Reference Vacuum), and vacuum decay (Reference Vacuum Deacy) 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. An approach that may be used to establish leak test critical parameters is described below:

A2.1.1 A population of control, non-leaking packages is exposed to various vacuum levels for lengthy Fill Time periods of 1 or more seconds to determine a Test Vacuum 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 (Fill Time). Once the Target Vacuum has been selected, a Reserve Vacuum value somewhat higher is selected to ensure that the Target Vacuum can be consistently achieved using control, no defect packages. When testing for package headspace gas leakage, Target Vacuum settings may range from +250 mbar to +500 mbar of pressure (absolute). Higher Target Vacuum conditions between 0 and +1 mbar are required to ensure liquid volatilization when testing for leaks partially or completely blocked by liquid.

A2.1.2 Equalization Time is selected by observing how long it takes for Test Vacuum to stabilize when testing control, nonleaking packages. When testing for leaks, Equalization time must be very short (typically <1 s) so the rapid rise in pressure resulting from liquid volatilization can be measured before saturation partial pressure is reached in the test system deadspace, at which point pressure rise no longer occurs..

A2.1.3 Test Time is selected by observing how long it takes for a significant pressure rise to occur in the test chamber when testing defective packages, or when testing controlled, non-leaking packages while simultaneously introducing a small, calibrated gas flow rate into the test chamber.

A2.1.4 Vacuum Decay critical parameters are selected by observing the Test Vacuum for a control package population. These baseline vacuum decay data are used to select the critical parameters of Reference Vacuum and Reference Vacuum Decay.

A2.1.5 A Pre-Test Vacuum Flush time is selected by varying this vacuum time and observing the subsequent Vacuum Decay readings obtained when testing control, nonleaking packages as compared to packages with the smallest defects. Optimum Pre-Test Vacuum Flush time will ensure consistent control package vacuum decay results which are noticeably lower than defective package vacuum decay results.

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.

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. Testing liquid-filled packages with and without defects is necessary to verify the ability of a leak test to identify liquid leaks.