Designation: F 2338 – 03

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.

1.2 *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} $Pa \cdot m^3 \cdot s^{-1}$.

1.3 *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

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.

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 according to manufacturer's specifications with non-defective lidding materials.

3.2.3 *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.

3.2.4 *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.

3.2.5 *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.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.60 on Medical Device Packaging.

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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.

3.3 *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, 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 tray or cup, or in the lid materials themselves (holes or cracks), or they may be found at the juncture of the seal made between the tray and the lid material (channel defects, tears, spotty or mottled seals). 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 package 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. The test chamber is connected to the vacuum decay test system. This system includes a vacuum source for establishing vacuum 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.