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# Standard Test Method for Measuring Package and Seal Integrity Using Helium as the Tracer Gas[1]

This standard is issued under the fixed designation F2391; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method includes several procedures that can be used for the measurement of overall package and seal barrier performance of a variety of package types and package forms, as well as seal/closure types. The basic elements of this method include:

1.1.1 Helium (employed as tracer gas),

1.1.2 Helium leak detector (mass spectrometer), and

1.1.3 Package/product-specific test fixtures.

1.1.4 Most applications of helium leak detection are destructive, in that helium needs to be injected into the package after the package has been sealed. The injection site then needs to be sealed/patched externally, which often destroys its saleability. Alternatively, if helium can be incorporated into the headspace before sealing, the method can be non-destructive because all that needs to be accomplished is to simply detect for helium escaping the sealed package.

1.2 Two procedures are described; however the supporting data in Section 14 only reflects Procedure B (Vacuum Mode). The alternative, Sniffer Mode, has proven to be a valuable procedure for many applications, but may have more variability due to exactly the manner that the operator conducts the test such as whether the package is squeezed, effect of multiple small leaks compared to fewer large leaks, background helium concentration, package permeability and speed at which the scan is conducted. Further testing to quantify this procedure's variability is anticipated, but not included in this version.

1.2.1 *Procedure A: Sniffer Mode*—the package is scanned externally for helium escaping into the atmosphere or fixture.

1.2.2 *Procedure B: Vacuum Mode*—the helium containing package is placed in a closed fixture. After drawing a vacuum, helium escaping into the closed fixture (capture volume) is detected. Typically, the fixtures are custom made for the specific package under test.

1.3 The sensitivity of the method can range from the detection of:

1.3.1 Large leaks—$10^{-2}$ Pa·m $^3$/s to $10^{-5}$ Pa·m$^3$/s ($10^{-1}$ cc/sec/atm to $10^{-4}$ cc/sec/atm).

1.3.2 Moderate leaks—$10^{-5}$ Pa·m $^3$/s to $10^{-7}$ Pa·m$^3$/s ($10^{-4}$ cc/sec/atm to $10^{-6}$ cc/sec/atm).

1.3.3 Fine leaks—$10^{-7}$ Pa·m $^3$/s to $10^{-9}$ Pa·m$^3$/s ($10^{-6}$ cc/sec/atm to $10^{-8}$ cc/sec/atm).

1.3.4 Ultra-Fine leak—$10^{-9}$ Pa·m $^3$/s to $10^{-11}$ Pa·m$^3$/s ($10^{-8}$ cc/sec/atm to $10^{-10}$ cc/sec/atm).

NOTE 1—Conversion from cc/sec/atm to Pa·m$^3$/s is achieved by multiplying by 0.1.

1.4 The terms large, moderate, fine and ultra-fine are relative terms only and do not imply the acceptability of any leak rate. The individual application dictates the level of integrity needed. For many packaging applications, only "large leaks" are considered unacceptable and the ability to detect smaller leaks is immaterial. All leak rates referred to in this method are based on conversion of actual conditions (based on partial pressure of helium) to one atmosphere pressure differential and standard temperature conditions.



1.5 The method may have applicability to any package type:

1.5.1 Flexible,

1.5.2 Semi-rigid, or

1.5.3 Rigid.

1.6 The sensitivities reported in the supporting data for this method pertain to the detectability of helium emanating from the sample and are not a function of the packaging form.

1.7 The method is not applicable to breathable or porous packaging.

1.8 The results obtained can be qualitative, semi-quantitative or quantitative depending on the procedure used.

1.9 Test fixture design is not within the scope of this method except to note that different designs will be needed for different applications (which have different package types and package integrity requirements). Furthermore, the fixture selection and design will be based on where the testing is to be conducted within the manufacturing process (in other words, quality control versus research).

[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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1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D996 Terminology of Packaging and Distribution Environments

D3078 Test Method for Determination of Leaks in Flexible Packaging by Bubble Emission

D4991 Test Method for Leakage Testing of Empty Rigid Containers by Vacuum Method

E432 Guide for Selection of a Leak Testing Method

E479 Guide for Preparation of a Leak Testing Specification (Withdrawn 2014)[3]

E493 Test Methods for Leaks Using the Mass Spectrometer Leak Detector in the Inside-Out Testing Mode

E498 Test Methods for Leaks Using the Mass Spectrometer Leak Detector or Residual Gas Analyzer in the Tracer Probe Mode

E499 Test Methods for Leaks Using the Mass Spectrometer Leak Detector in the Detector Probe Mode

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

E1603 Test Methods for Leakage Measurement Using the Mass Spectrometer Leak Detector or Residual Gas Analyzer in the Hood Mode

F17 Terminology Relating to Flexible Barrier Packaging

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

2.2 *Other Documents:*

Principal author L. Kirsch, et al - (shown in reference Appendix X1 as literature references 1, 2, 3 and 5)

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## 3. Terminology

3.1 *General Term Definitions*—For definitions used in this standard see Terminology D996, Terminology F17 and Terminology F1327.

3.2 *Specific Term Definitions:*

3.2.1 *actual helium leak rate (AHLR)*—Measured helium leak rate (MHLR) signal level adjusted to a driving force of 100 % concentration at 101 KPa (1.0 atmosphere), absolute.

3.2.2 *breathable/porous packaging*—Packages, in whole or in part, that intentionally allow gases/vapors to flow freely into and out of the package. (See also Terminology F1327)

3.2.3 *fine leaks*—For the purpose of this test method, leaks that exhibit gas/vapor leak rates between $1\times10^{-7}$ Pa·m$^3$/s to $10^{-9}$ Pa·m$^3$/s ($1\times10^{-6}$ cc/sec/atmosphere to $1\times10^{-8}$ cc/sec/atmosphere).

3.2.4 *flexible packaging*—Packages (typically, pouches, sachets, and bags) constructed of materials that are readily bendable. (See also Terminology Method F17)

3.2.5 *impermeable packaging*—Packages constructed of materials (typically metal or glass) that prevent gases/vapors from flowing into or out of the package.

3.2.6 *large leaks*—For the purpose of this test method, leaks that exhibit gas/vapor leak rates between $1\times10^{-2}$ Pa·m$^3$/s to $1\times10^{-5}$ Pa·m $^3$/s ($1\times10^{-1}$ cc/sec/atm to $1\times10^{-4}$ cc/sec/atmosphere).

3.2.7 *measured helium leak rate (MHLR)*—Helium signal level obtained based on the actual helium concentration in the package.

3.2.8 *moderate leaks*—For the purpose of this test method, leaks that exhibit gas/vapor leak rates between $1\times10^{-5}$ Pa·m$^3$/s to $10^{-7}$ Pa·m $^3$/s ($1\times10^{-4}$ cc/sec/atmosphere to $1\times10^{-6}$ cc/sec/atmosphere).

3.2.9 *outgassing*—The release of adsorbed, absorbed or physically trapped gas from a surface of structure.

3.2.10 *pass/fail criterion*—The predetermined AHLR above which the package being tested is considered defective and, therefore, unacceptable.

3.2.11 *permeable packaging*—Packages, in whole or in part, that allow gases/vapors to flow into and out of a package via diffusion controlled process.

3.2.12 *semi-rigid packaging*—Packages (typically, thermoformable, or cold-formable materials) that are formed into blisters or trays, with associated lidding materials applied as the closure means.

3.2.13 *ultra fine leaks*—For the purpose of this test method, leaks that exhibit gas/vapor leak rates between $1\times10^{-9}$ Pa·m$^3$/s to $1\times10^{-11}$ Pa·m $^3$/s ($1\times10^{-8}$ cc/sec/atmosphere to $1\times10^{-10}$ cc/sec/atmosphere).

3.2.14 *virtual leak*—A source of detectable tracer gas other than from a defect of the seal or package. Such a virtual leak may be the result of membrane permeability, surface desorption or release of trapped gas.

## 4. Summary of Test Procedures

4.1 There are two basic test procedures contained in this test method:

4.1.1 *Procedure A*—Sniffer Mode.

4.1.2 *Procedure B*—Vacuum Mode.

4.2 Both of these test procedures require the package under test to have helium at some measurable level on the side of the package opposite the leak detector sensor (typically, the inside of the package). If the package cannot, or should not be sealed with helium inside, the test fixture used for that particular test

[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.

needs to provide a means of helium introduction at the appropriate location and the appropriate time in the test cycle. The one exception is a package with a gross leak for which a variation of the helium pressurized "back-filling" or "soaking" technique may be applicable. In all cases helium, at as high a concentration as practicable, must be present on one side of the package/seal barrier element.

4.3 To quantify the leak rate level of a given package, or package seal, the partial pressure driving force of the helium must be known. Therefore, an important part of the process of conducting a leak rate test is the determination of the concentration of helium at one atmosphere (absolute pressure) present during the test. Generally speaking, some type of calibrated residual gas analyzer (RGA) device will need to be utilized for this step.

4.4 The MHLR (measured helium leak rate) values will be determined based on a comparison to the calibration, reference standard employed. It is subsequently adjusted to an AHLR (actual helium leak rate), which is based on the actual package helium partial pressure (see 4.5).

4.5 If appropriate, the AHLR value for the package under test can be compared to the pre-established Pass/Fail criterion for that specific product/package to ascertain acceptability (per established specification requirements).

## 5. Significance and Use

5.1 The vacuum, bubble test method, as described in Test Method D3078, and various other leak detection methods described elsewhere (Test Method D4991, Guide E432, Guide E479, Test Method E493, Test Method E498, Test Method E499, and Test Method E1603) have been successfully used widely in various industries and applications to determine that a given package is or is not a "leaker." The sensitivity of any selected leak test method has to be considered to determine its applicability to a specific situation.

5.2 The procedures presented in this test method allow the user to carry out package and seal integrity testing with sufficient sensitivity to quantify seals in the previously defined moderate to very fine seal ranges.

5.3 By employing seal-isolating leak testing fixtures, packages constructed of various materials can be tested in the full range of seal performance requirements. Design of these fixtures is beyond the scope of this method.

5.4 These seal/package integrity test procedures can be utilized as:

5.4.1 A design tool,

5.4.2 For tooling qualification,

5.4.3 Process setup,

5.4.4 Process validation tool,

5.4.5 Quality assurance monitoring, or

5.4.6 Research and development.

## 6. Interferences

6.1 The introduction of the helium tracer gas to the non-sensor side of the package (typically the inside) can be done either before or after sealing.

6.2 Some helium may be present in the testing environment which may interfere with results. Care must be taken to eliminate background helium with ventilation, location of supply cylinders, proper sample isolation fixturing or other means.

6.3 When attempting to detect very small leaks, care must be taken to eliminate, minimize, or compensate for false readings from "virtual leak" sources, particularly trapped helium in seal areas.

6.4 The permeation of the package by helium does not indicate a leak. Care must be taken to understand the level of permeation to prevent misinterpretation of results. Similarly, some materials may absorb helium and yield false results when tested. Outgassing of these materials may greatly increase test time.

6.5 These procedures, particularly when detecting moderate to very fine leaks, should be carried out using calibrated external leak standards.

6.6 Physical/mechanical constraints are generally required for flexible and semi-rigid packages to avoid vacuum-induced seal failures. Properly constrained packages can mean the difference between success and failure in carrying out the test procedure.



## 7. Apparatus

7.1 A helium leak detector (mass spectrometer). An oil-free vacuum system is recommended with hard vacuum test port and sniffer probe attachment (as appropriate for a specific application) for those applications where the testing area needs to be maintained as a clean environment, or where the release of vacuum pump oil could lead to product contamination, or both.

7.2 External calibrated leaks (calibrated within the last 12 months; 6 months is recommended). At least three ranges should be covered depending on the application; typically $1\times10^{-6}$, $1\times10^{-7}$ and $1\times10^{-8}$ cc/s/atm. Alternatively, more calibrated leaks may be used.

7.3 A vacuum chamber, with custom-design constraints that are package-specific (sniffer mode testing may not require a vacuum chamber).

7.4 A headspace analyzer device for measuring the partial pressure of (concentration at 1 atm pressure) helium in samples.

7.5 The method to introduce helium into the package needs to be developed specifically for the package under test. Techniques and devices that have been successfully employed include:

7.5.1 Pre-filling of packages using an on-line flooding fixture (helium introduced to package headspace prior to sealing).

7.5.2 Post-filling of packages by injection of helium into the sealed package. A fine gage syringe needle and flow-controlled helium gas supply, followed by sealing of the puncture site has been found to work well.