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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)

Principal author L. Nguyen, et al - (shown in reference appendix I at literature reference 4)

Co-authors include C. Moeckly, L. Nguyen, R. Gerth, W. Muangsiri, R. Scheire, D. M. Guazzo, L. Kirsch, G. Schmitt, A. Kirsch, M. Koch, T. Wertli, M. Lehman and G. Schramm.

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 1×10^{-7} Pa·m^3/s to 10^{-9} Pa·m^3/s (1×10^{-6} cc/sec/atmosphere to 1×10^{-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 1×10^{-2} Pa·m^3/s to 1×10^{-5} Pa·m ^{3}/s (1×10^{-1} cc/sec/atm to 1×10^{-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 1×10^{-5} Pa·m^3/s to 10^{-7} Pa·m ^{3}/s (1×10^{-4} cc/sec/atmosphere to 1×10^{-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 1×10^{-9} Pa·m^3/s to 1×10^{-11} Pa·m ^{3}/s (1×10^{-8} cc/sec/atmosphere to 1×10^{-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 1×10^{-6}, 1×10^{-7} and 1×10^{-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.

7.6 An enclosure for sniffer mode testing seals/packages for moderate, fine, or ultra-fine leaks in a lowered helium background environment may be found necessary to reduce inference of background helium.

7.7 A data acquisition and analysis device is optional, but recommended, for recording, or calculating, or both the measured helium leak rates (MHLR), helium partial pressure and the actual (normalized) helium leak rate (AHLR).

8. Instrument Calibration

8.1 *Sensor Calibration*—Most contemporary helium leak detector units have a built-in calibrated reference leak that is used to calibrate the mass spectrometer sensor incorporated into the leak detector. Generally, the accelerating voltage and emission currents settings are automatically adjusted as needed to have the sensor agree with the calibrated reference leak. This calibration procedure should be carried out frequently, typically once a day.

8.2 *Test Fixture Calibration*—

8.2.1 For Procedure A (Sniffer Mode), external calibration leaks, with custom housing assemblies, can be used.

8.2.2 For Procedure B (Vacuum Mode), the sample chamber and headspace analyzer fixtures must be designed to optimize sensitivity. Inclusion of calibration leaks or calibrated helium gas mixtures can be used.

8.3 *Total System Calibration*—For calibration of the full system (when operating in the vacuum mode), externally calibrated leaks are sequentially mounted on the vacuum test port of the leak detector.

9. Reagents and Materials

9.1 *Helium (nominally 100 %) Gas*—supply cylinder and regulator. Flow meter is optional but recommended.

9.2 *Nitrogen (nominally 100 %) Gas*—supply cylinder and regulator. Flow meter is optional, but recommended.

9.3 *He/N_2 calibrated gas mixtures; typically, 75 % helium & 50 % helium*—supply cylinder and regulator (flow meter, optional, but recommended).

9.4 Pressure-sensitive adhesive aluminum tape can be used effectively to seal the site where helium was injected into the package headspace (post-filling) when post sealing injection of helium is used.

10. Sample Preparation

10.1 *Pre-filling of Packages*—The preferred option is to introduce the helium tracer gas immediately prior to the sealing operation. A high concentration (50 % –100 %) of helium within the headspace of the package as each package is closed provides the source of helium for subsequent detection of leakage pathways present in the package walls or seal areas.

10.2 *Post-filling of Packages*—When packages cannot be pre-filled with helium, helium gas flushing through puncture sites (inlet and outlet sites) can be used, followed by sealing of these puncture sites. Care must be taken to maintain a very slight or, preferably, zero headspace pressure in each package.

11. Procedures and Calculations

11.1 *Procedure A—Sniffer Mode Gas/Vapor Barrier Seal and Package Integrity Test for Permeable and Impermeable Packaging*

11.1.1 Start the helium leak detector unit and allow sufficient warm-up time before sensor calibration (per manufacturer's instructions).

11.1.2 Carry out the sensor (mass spectrometer) calibration (per manufacturer's instructions).

11.1.3 Attach the sniffer probe to the leak detector and set the detector for sniffer probe mode.

11.1.4 For moderate, fine, or ultra-fine leaks, test the packages/seals in an enclosure with a N_2 atmosphere (to minimize interference from atmospheric helium).

11.1.5 Bring the sniffer probe gas collection tip in close proximity (approximately 1⁄16 – 1⁄8 in.) to the area (portion of a seal or portion of a package sidewall) to be leak checked.

11.1.6 Scan (manually or automatically) over the package with the sniffer probe at approximately 1⁄8 inch of travel per second.

11.1.7 Note areas, if any, where the measured leak rate exceeds the pre-determined pass/fail level. Although difficult to insure complete compliance, the final leak rate for a given package should be recognized as the sum of all individual leaks in the package.

11.1.8 Optionally, determine the headspace concentration of helium and calculate the AHLR (actual helium leak rate) by dividing the MHLR by the helium partial pressure.

11.1.9 Record the data and results (See Section 12.)

11.2 *Procedure B—Vacuum Mode Gas/Vapor Barrier Seal & Package Integrity Test for Permeable & Impermeable Packaging*

11.2.1 Start the helium leak detector unit and allow sufficient warm-up time before sensor calibration (per manufacturer's instructions).

11.2.2 Carry out the sensor (mass spectrometer) calibration (per manufacturer's instructions).

11.2.3 Carry out the system calibration procedure using the three external calibrated leaks sequentially mounted on the leak detector test port.

11.2.4 Mount the vacuum chamber, or the headspace analyzer fixture, or both on the leak detector test port.

11.2.5 Carry out leak rate calibration/validation of the vacuum chamber using custom calibrated leaks (optional).

11.2.6 Carry out a helium concentration calibration of the headspace analyzer fixture if using the mass spectrometer as the sensor using the calibrant gases.

11.2.7 *Specific Test Vacuum Mode:*

11.2.7.1 Determine the indicated or measured helium leak rate (MHLR) of individual samples placed within the specifically designed test fixture.

11.2.7.2 Measure the actual helium concentration contained within each package (partial pressure).

11.2.7.3 Calculate the actual helium leak rate (AHLR) for each individual package by dividing the MHLR by the individual package's helium partial pressure.

11.2.7.4 Compare the AHLR with the pre-established pass/fail level for the product/package being tested. Any packages which exceed the pre-established pass/fail level may be subjected to further examination as appropriate.

11.2.7.5 Record the data and results. (See Section 13.)

11.2.8 *Average Test Vacuum Mode*

11.2.8.1 Measure the actual helium concentration contained within the headspace (partial pressure) for a pre-selected set of samples and calculate the average value of the set of samples.

11.2.8.2 Determine the indicated or measured helium leak rate (MHLR) of individual samples placed within the specifically designed test fixture.

11.2.8.3 Calculate the actual helium leak rate (AHLR) for each individual package by dividing the MHLR by the average helium partial pressure.

11.2.8.4 Compare the AHLR with the pre-established pass/fail level for the product/package being tested. Any packages that exceed the pre-established pass/fail level may be subjected to further examination as appropriate.

11.2.8.5 Record the data and results. (See Section 13.)

12. Additional Calculations

12.1 A correction in the certified leak standard used may be necessary as the calibration leak ages. The partial pressure of helium contained within the reservoir slowly changes with time and, therefore, the actual leakage changes. Follow the manufacturer's instructions to determine the actual leak rate of the calibration standard. A temperature correction may also be necessary and, again, the manufacturer's instructions are to be followed.

13. Report

13.1 The Report shall include the following:

13.1.1 A statement indicating that the tests were carried out according to this test method noting any exceptions to the specific procedures or manufacturer's instruction. Other factors that may affect results such as temperature are reported as well.

13.1.2 The Pass/Fail (P/F) values, MHLR and AHLR values should be reported in SI units of Pa·m^3/s (or cc/sec/atm as appropriate). The units of helium gas concentration shall typically be reported in atmospheres (in decimal form).

13.1.3 The specific test procedure, or test procedures employed.

13.1.4 The serial numbers, the calibration values and most recent calibration dates for all the calibration standards used.

13.1.5 Copies of any software-generated data sheets, or reports produced during the testing.

14. Precision and Bias

14.1 *Precision*—While the precision of these test methods vary with each instrument and the sensitivity level of the test, the leak tester level of precision generally stated by the manufacturers is ± 30 %. This is a conservative (worst case) estimate. The same samples have been tested at three different laboratories and have resulted in calculated values for intra-laboratory precision of ± 15 % and inter-laboratory precision of ±22 %.

14.2 *Bias*—The bias of the helium leak test will, generally, be equal to that of the standard leaks used for calibration of the system when conditions are the same as the system operating conditions. Typically the NIST-traceable calibration leak standards used for helium leak detectors have a precision rating of ±10 %.

14.3 A description of a 2002–2003 interlaboratory test, which was conducted in accordance with Procedure B, is included as Appendix X2. For further insight into interlaboratory testing, see Practice E691.

15. Keywords

15.1 blister packaging; bottles; cartridges; flexible packaging; gas barrier performance; helium leak test; impermeable packaging (and materials); integrity monitoring; mass spectrometer; mass spectrometer leak test; mass spectrometer sensor; pass/fail criteria; pass/fail levels; package integrity monitoring; package integrity test; permeable packaging (and materials); pouch packaging; seal integrity monitoring; seal integrity test; sniffer probe leak test; sterility barrier performance; vacuum leak test; vapor barrier performance; rigid packaging; vials

APPENDIXES

(Nonmandatory Information)

X1. CITED TECHNICAL LITERATURE

1. Lee E. Kirsch, Lida Nguyen and Craig S. Moeckly, "Pharmaceutical Container/Closure Integrity I: Mass-Spectrometry-based helium Leak Rate Detection For Rubber-Stoppered Vials," *PDA Journal of Pharmaceutical Science and Technology* **51**(5), 1997.

2. Lee E. Kirsch, Lida Nguyen, Craig S. Moeckly and Ronald Gerth " Pharmaceutical Container/Closure Integrity II: The Relationship Between Microbial Ingress and helium Leak Rates In Rubber-Stoppered Vials," *PDA Journal of Pharmaceutical Science and Technology* **51**(5), 1997.

3. Lee E. Kirsch, Lida Nguyen and Ronald Gerth "Pharmaceutical Container/Closure Integrity III: Validation of the helium Leak Rate Method," *PDA Journal of Pharmaceutical Science and Technology* **51** (5), 1997.

4. Lida Nguyen, Walasiri Muangsiri, Raymond Scheire, Dana Morton Guazzo and Lee E. Kirsch, "Pharmaceutical Container/Closure Integrity IV: Development of an Indirect Correlation Between Vacuum Decay Leak Measurement and Microbial Ingress," *PDA Journal of Pharmaceutical Science and Technology* **53** (4), 1999.

5. Lee E. Kirsch, Lida Nguyen, Ann Marie Kirsch, Garry Schmitt, Marcel Koch, Toni Wertli, Martin Lehman and Gerhard Schramm, "Pharmaceutical Container/Closure Integrity V: An Evaluation of the WILCO "LFC" Method for Leak Testing Pharmaceutical Glass-Stoppered Vials," *PDA Journal of Pharmaceutical Science and Technology* **53**(5), 1999.

X2. INTERLABORATORY TEST DESCRIPTION

INTRODUCTION

A Round Robin Testing Program, in support of Helium Leak Testing (Package/Seal Integrity Testing) Method, was conducted in 2002-2003. What is provided below is a summary of a statistical analysis of the data generated by 12 operators on three Helium leak detectors and four participating laboratory sites. A total of 720 measures of the leak rate of samples were made. More detailed description of the test and all individual data are available from ASTM as a research report.

The testing protocol is described below in outline form:

X2.1 Helium Leak Rate (HeL/R) Test Method Description

X2.1.1 Cold-Form-Aluminum-Foil blister packaging with a polyester/foil heat-sealed lidding material constitutes the samples that were tested.

X2.1.2 Helium gas was introduced via an 18 gauge syringe needle through a puncture site in the lidding portion of the individual samples; followed by the application of an aluminum foil sealing tape over the puncture site (high quality seal integrity samples), or the application of a more permeable, Scotch sealing tape over the puncture site (lesser quality packages—"defectives"). The latter samples represent packages that would be considered "leakers" based on the somewhat arbitrarily defined pass/fail criteria defined below.

X2.1.3 All samples were prepared at the LDA Laboratories and hand-delivered to the testing site/s. The method of tracer gas (Helium) filling has been standardized for consistency of achieved headspace concentration.

X2.1.4 The Helium gas concentration levels of representative samples were measured prior to preparation of the test samples and the average value of Helium concentration was supplied to each tester and test site.

X2.1.5 A standard format data entry form was provided to each participant (tester) and to each testing location (testing site/laboratory).

X2.1.6 The Pass/Fail (P/F) Criterion for this testing program was been set at 1×10^{-7} cc/second (@ 100 % Helium concentration within the package).

X2.1.7 The Helium Leak Tester Unit, or Units, used in the program were calibrated using three NIST-Traceable external calibrated leaks (in the range of 1×10^{-6}, 1×10^{-7} and 1×10^{-8} cc/sec/atmosphere).

X2.1.8 Each instrument underwent a three-point calibration/re-validation procedure at the start of a given test set.

X2.1.9 The Helium Leak Rate (HeL/R) readings for all samples tested were noted from the alphanumeric digital display of the detection system. The Measured Helium Leak Rate (MHLR) readings were recorded 30 seconds after the test cycle went into the fine leak mode. Method B of this standard method was followed.

X2.1.10 The Actual (normalized) Helium Leak Rate (AHLR) Values were determined by dividing the MHLR by the average headspace Helium concentration (in decimal form).

X2.1.11 The AHLR Values were compared with the P/F Criterion of 1×10^{-7} and all samples $\leq 1\times10^{-7}$ are designated "Pass" and all samples $> 1\times10^{-7}$ are designated "Fail."

X2.1.12 The lot size for both the aluminum foil tape resealed samples and the scotch tape resealed for each tester was ten (10). Three repeat measurements were made on each sample.

X2.2 Test Data (Preliminary)

X2.2.1 The accompanying tables present a summary of the data generated. A total of four (4) "sites/instruments" and twelve (12) individuals participated. The four (4) sites/instruments involved in the program were designated as "Site/Instrument" Location L, W, P & M, respectively. The individual testers were designated by number such as: L-1, L-2, L-3, etc.

X2.3 Commentary

X2.3.1 These data/results show excellent agreement for both inter-laboratory and intra-laboratory testing.

X2.3.2 The samples defined as defective were differentiated from the non-defective perfectly (240 were correctly identified out of a total of 240 samples tested). Each of these samples was tested three times making 720 correct measures.

TABLE X2.1 Results (Aluminum Foil Tape Sealed—“Non-Defective”)

Site/Tester	MHLR 10-Sample Avg cc/second	% Helium 20-Sample Avg in decimal form	AHLR 10-Sample Avg cc/sec/atm	P/F (Comments)
Site L				
L-1	2.0 ± 0.13 × E-8	0.62	3.2 ± 0.21 × E-8	Pass (10/10)
L-2	1.8 ± 0.14 × E-8	0.62	3.0 ± 0.22 × E-8	Pass (10/10)
L-3	1.7 ± 0.17 × E-8	0.62	3.5 ± 0.27 × E-8	Pass (10/10)
Site Total	1.8 ± 0.15 × E-8	0.62	3.2 ± 0.22 × E-8	Pass (30/30)
Site W				
W-1	2.0 ± 0.11 × E-8	0.62	3.2 ± 0.18 × E-8	Pass (10/10)
W-2	1.9 ± 0.23 × E-8	0.62	3.1 ± 0.37 × E-8	Pass (10/10)
W-3	2.1 ± 0.21 × E-8	0.62	3.6 ± 0.34 × E-8	Pass (10/10)
Site Total	2.0 ± 0.18 × E-8	0.62	3.3 ± 0.27 × E-8	Pass (30/30)
Site P				
P-1	2.1 ± 0.20 × E-8	0.65	3.2 ± 0.32 × E-8	Pass (10/10)
P-2	1.9 ± 0.19 × E-8	0.62	3.1 ± 0.31 × E-8	Pass (10/10)
P-3	2.1 ± 0.09 × E-8	0.62	3.4 ± 0.15 × E-8	Pass (10/10)
Site Total	2.0 ± 0.16 × E-8	0.63	3.5 ± 0.25 × E-8	Pass (30/30)
Site M				
M-1	2.6 ± 0.19 × E-8	0.60	4.3 ± 0.27 × E-8	Pass (10/10)
M-2	1.8 ± 0.13 × E-8	0.60	3.0 ± 0.31 × E-8	Pass (10/10)
M-3	2.3 ± 0.16 × E-8	0.60	3.8 ± 0.21 × E-8	Pass (10/10)
Site Total	2.2 ± 0.16 × E-8	0.60	3.7 ± 0.27 × E-8	Pass (30/30)
Totals	2.0 ± 0.18 × E-8	0.62	3.2 ± 0.30 × E-8	Pass (120/120)

TABLE X2.2 Results (Permeable—Scotch Tape Sealed—“Defective”)

Site/Tester	MHLR 10-Sample Avg cc/second	% Helium 20-Sample Avg in decimal form	AHLR 10-Sample Avg cc/sec/atm	P/F (Comments)
Site L				
L-1	2.5 ± 0.28 × E-6	0.59	3.8 ± 0.21 × E-6	Fail (10/10)
L-2	1.8 ± 0.18 × E-6	0.59	3.1 ± 0.22 × E-6	Fail (10/10)
L-3	2.2 ± 0.19 × E-6	0.59	3.7 ± 0.27 × E-6	Fail (10/10)
Site Total	2.2 ± 0.21 × E-6	0.59	3.5 ± 0.37 × E-6	Fail (30/30)
Site W				
W-1	1.6 ± 0.18 × E-6	0.59	2.7 ± 0.28 × E-6	Fail (10/10)
W-2	1.7 ± 0.10 × E-6	0.59	2.9 ± 0.37 × E-6	Fail (10/10)
W-3	2.2 ± 0.20 × E-6	0.59	3.9 ± 0.34 × E-6	Fail (10/10)
Site Total	1.8 ± 0.16 × E-6	0.59	3.2 ± 0.31 × E-6	Fail (30/30)
Site P				
P-1	1.8 ± 0.12 × E-6	0.56	3.2 ± 0.32 × E-6	Fail (10/10)
P-2	1.6 ± 0.14 × E-6	0.59	2.7 ± 0.31 × E-6	Fail (10/10)
P-3	2.6 ± 0.11 × E-6	0.59	4.4 ± 0.18 × E-6	Fail (10/10)
Site Total	2.1 ± 0.12 × E-6	0.58	3.4 ± 0.28 × E-6	Fail (30/30)
Site M				
M-1	3.7 ± 0.24 × E-6	0.57	6.4 ± 0.37 × E-6	Fail (10/10)
M-2	2.3 ± 0.18 × E-6	0.57	4.0 ± 0.31 × E-6	Fail (10/10)
M-3	2.6 ± 0.16 × E-6	0.57	4.6 ± 0.29 × E-6	Fail (10/10)
Site Total	2.9 ± 0.22 × E-6	0.57	5.0 ± 0.34 × E-6	Fail (30/30)
Totals	2.4 ± 0.18 × E-6	0.58	3.8 ± 0.30 × E-6	Fail (120/120)

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