Designation: F2095 – 07 (Reapproved 2013)

Standard Test Methods for Pressure Decay Leak Test for Flexible Packages With and Without Restraining Plates[1]

This standard is issued under the fixed designation F2095; 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 These test methods cover the measurement of leaks in nonporous film, foil, or laminate flexible pouches and foil-sealed trays, which may be empty or enclose solid product. If product is enclosed, seals or surfaces cannot be in contact with water, oils, or other liquid.

1.2 These test methods will detect leaks at a rate of 1×10^{-4} sccs (standard cubic centimetres per second) or greater, in flexible packages. The limitation of leak rate is dependent on package volume as tested.

1.3 The following test methods are included:

1.3.1 *Test Method A*—Pressure Decay Leak Test for Flexible Packages Without Restraining Plates

1.3.2 *Test Method B*—Pressure Decay Leak Test for Flexible Packages With Restraining Plates

1.4 These test methods are destructive in that they require entry into the package to supply an internal pressure of gas, typically air or nitrogen, although other gases may be used. The entry connection into the flexible package must be leak-tight.

1.5 For porous packages, see 9.3.

1.6 *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]

D4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

F17 Terminology Relating to Flexible Barrier Packaging

2.2 *Other Document:*

ANSI/AAMI/ISO 11607–1:2006 Packaging for Terminally Sterilized Medical Devices—Part 1: Requirements for Materials, Sterile Barrier Systems, and Packaging Systems[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *integrity*—the unimpaired physical condition of the package. This implies that there are no leaks in the seals or body materials.

3.1.2 *leak*—See Terminology F17.

3.1.3 *nonporous*—types of materials that are not purposely designed to transfer gases through their matrix.

3.1.4 *restraining plates*—plates of rigid material, for example, aluminum, that are used to restrict the movement of the package during inflation.

3.1.5 *seal*—See Terminology F17.

3.1.6 *standard cubic centimetre per second (sccs)*—the flow rate of a gas (air) at standard conditions of 20°C (68°F) and 101.3 kPa (14.7 psig) (1 atmosphere or 760 mm Hg).

3.1.6.1 *Discussion*—Conditions may be varied depending on the source of data. Always check the definition being used.

4. Summary of Test Method

4.1 Detection of leak paths in flexible packages that have nonporous material surfaces and seals can be accomplished by pressurization of the package to a fixed pressure, shutting off the pressure and connecting a pressure transducer. Observed changes in pressure indicate the presence of leakage paths in the package seals or pinholes in the surfaces. This leak may be represented in decay pressure units or calculated leak rate units. To accomplish this technique, a leak-tight measuring

[1] These test methods are under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packaging and are the direct responsibility of Subcommittee F02.40 on Package Integrity.

Current edition approved Aug. 1, 2013. Published September 2013. Originally approved in 2001. Last previous edition approved in 2007 as F2095 – 07. DOI: 10.1520/F2095-07R13.

[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

path must be available between the package interior volume and the pressure transducer (see Fig. 1).

NOTE 1—The coating used on porous barrier films will mask defects (pin-holes) in/through the porous material but not defects in the seals.

4.2 Restraining plates may be used to limit the volume of the pressurized package. Because the sensitivity of these test methods is dependent in part on the internal volume of the package, the effect of restraining plate use is to increase the sensitivity of the test (see Fig. 2). See Appendix X1 for further discussion of the effects of restraining plates on these test methods.

5. Significance and Use

5.1 These test methods provide a rapid, simple to apply method to detect small leaks in flexible package seals or walls at the leak rate level of greater than 1×10^{-4} sccs, thus providing a measure of package integrity. Porous barrier film packages made non-porous with an impermeable film forming coating may demonstrate lateral leakage through the barrier material. Verification of leakage differences from background leakage must be included in validation methods. The use of calibrated hole sizes or orifices may be appropriate to determine leakage sensitivity or barrier integrity for these materials.

5.2 While theoretical leak rate sensitivity can be established by calculation, the test measurement is in pressure units and the measuring instrument must be calibrated, certified, and verified with these units.

5.3 The pressure decay method of leak testing is a physical measure of package integrity. When testing medical packaging which must conform to ISO 11607–1: 2006 standards, it may necessary to verify the results of the pressure decay test method with other sterile package integrity test methods.

5.4 Test Method A allows packages to be pressurized without restraint. In Test Method A the pouch, tray, or other type package will contain a volume of air defined by its mechanical configuration and its ability to resist internal pressure applied. This test method requires that the package reach a stable volume configuration (stop stretching) to make a measurement.

5.5 Test Method B allows the use of rigid restraining plates against the walls of the package to limit its volume and stabilize the package volume.

6. Apparatus

6.1 *Test Method A:*

6.1.1 A measuring instrument that provides the following:

6.1.1.1 A means to detect pressure changes with sufficient sensitivity to achieve theoretical leak rates in the package specification;

6.1.1.2 Automatic timer controls to pressurize the package to a preset pressure, hold the pressure for a set time, and provide a time period during which pressure change data can be taken;

6.1.1.3 A means to set pressure;

6.1.1.4 A means of holding and displaying the pressure change inside the package at the end of the test cycle;

6.1.1.5 A means (optional) to set pressure decay limits for a test method and alert the operator if the limit is exceeded.

6.1.2 A means to enter the package in a leak tight manner so that an inflation pressure can be applied to the package and changes in internal pressure can be sensed.

NOTE 2—It is important to verify the leak integrity of the entry means so that it does not contribute to the pressure changes sensed during testing.

6.2 *Test Method B—Using Restraining Plates:*

6.2.1 The measuring instrument shall have the characteristics described in 6.1.1.1-6.1.1.5.

6.2.2 Parallel, rigid plates are required. An ability to adjust plate separation is desirable. The surface of the plates should provide limited porosity in order to prevent blocking of pinhole leaks in the walls (see Fig. 2).

NOTE 3—Several techniques have been used to provide a means to prevent blocking or lowering of the leak rate in package material walls in

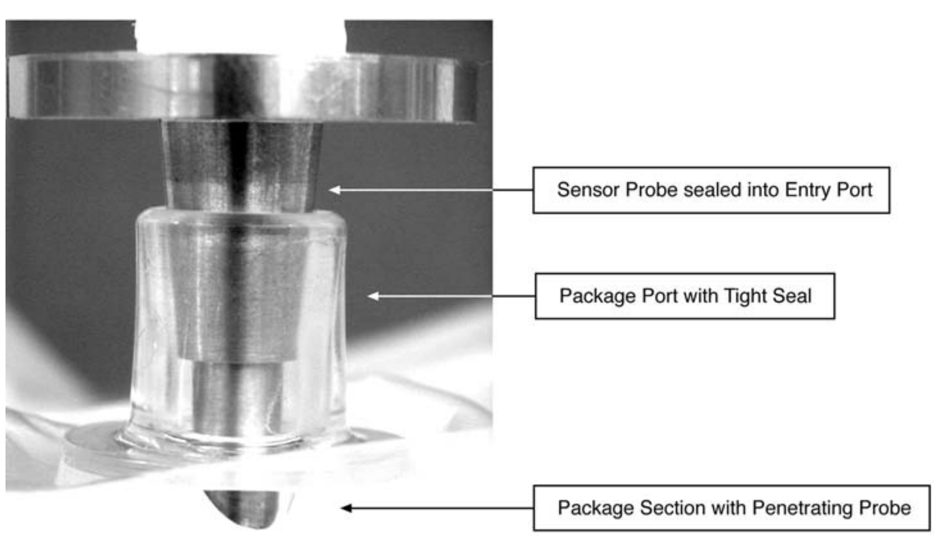

FIG. 1 Leak-Tight Entry System

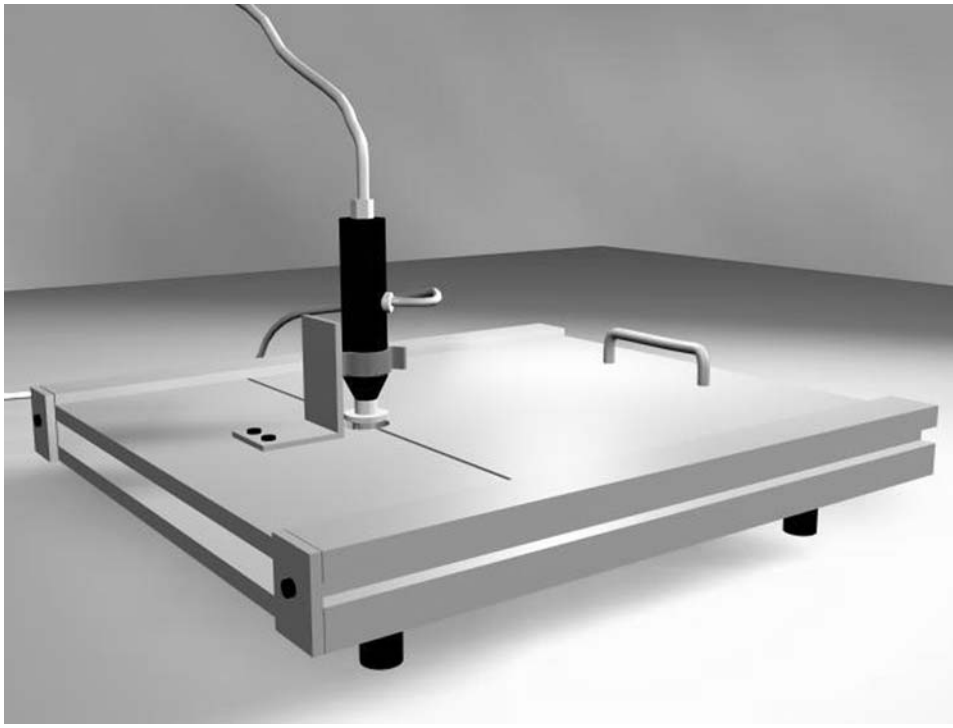

FIG. 2 Restraining Fixture with Leak-Tight Entry System

contact with the plates. These techniques include the use of semi-porous plastic, scoring of plate surfaces and use of screen-type materials.

6.2.3 A means to enter the package in a leak-tight manner so that an inflation pressure can be applied to the package and changes in internal pressure can be sensed.

NOTE 4—It is important to verify the leak integrity of the entry means so that it does not contribute to the pressure changes sensed during testing.

7. Sampling

7.1 The sample size is chosen to permit an adequate determination of representative performance.

7.2 Sample identification should be made prior to testing to allow the operator to refer to specific test samples, if necessary. Record information such that test results and anomalies are identifiable back to the individual specimens.

8. Conditioning

8.1 Package samples should be conditioned to obtain the same temperature conditions as exist for the test apparatus. Since measured pressure change is also a function of temperature, then the samples must be at a stable temperature. Most testing will occur at standard laboratory conditions of 23 ± 2°C (73 ± 4°F) and 50 ± 5 % relative humidity. Other conditions should be recorded at the time of the test.

NOTE 5—As seen in the combined gas laws, the pressure change is a function of temperature. Test packages and the test medium (air) should be at similar temperatures.

9. Procedure

9.1 *Test Method A—No Restraining Plates:*

9.1.1 *Package Preparation*—The package may be tested with or without the product enclosed. To maximize sensitivity of the test, the smallest internal volume of the package is desired.

9.1.2 *Instrument Preparation* (see Annex A1 for information on determining appropriate test parameters):

9.1.2.1 Select and set the test pressure.

9.1.2.2 Select and set the timers for charge (pressurization), settle (stabilization), and test (data taking period).

9.1.2.3 Select and set pressure decay limits (if available).

9.1.3 Attach the inflation probe (supply and sensor) to the instrument.

9.1.4 Attach the leak-tight entry device and inflation probe sensor to the package (see Fig. 1).

9.1.5 Begin the test by activating the timer controls and valves to inflate, hold, and measure the test pressure inside the package.

9.1.6 Observe the pressure decay at the end of the test time period, and note if the pressure decay limit has been exceeded.

NOTE 6—Choice of times depends on package variables and leak rate requirements. For example, small changes in initial test pressure may occur from flexible package stretch, thus slightly increasing its volume (decreasing its pressure) or from fixture contact or the expanding gas medium. Increased stabilization time will allow these effects to become stable before the test data period begins. Test times are selected based on required leakage rates or pressure decay criteria along with the package volume. See Annex A1 for further discussion.

9.2 *Test Method B—With Restraining Plates:*

9.2.1 *Package Preparation*—The package may be tested with or without the product enclosed. To maximize sensitivity of the test, the smallest internal volume of the package is desired. To achieve the minimum volume, the smallest gap between restraining plates is advisable.

9.2.2 *Instrument Preparation* (see Annex A1 for information on determining appropriate test parameters):

9.2.2.1 Select and set the test pressure.

9.2.2.2 Select and set the timers for charge (pressurization), settle (stabilization), and test (data taking period).

9.2.2.3 Select and set pressure decay limits (if available).

9.2.3 Attach the inflation probe (supply and sensor) to the instrument.

9.2.4 Attach the leak-tight entry device and inflation probe sensor to the package (Fig. 1).

9.2.5 Enclose the package and probe in the restraining fixture.

9.2.6 Begin the test by activating the timer controls and valves to inflate, hold, and measure the test pressure inside the package.

9.2.7 Observe the pressure decay at the end of the test time period and note if the pressure decay limit has been exceeded.

NOTE 7—Choice of times depends on package variables and leak rate requirements. For example, small changes in initial test pressure may occur from flexible package stretch, thus slightly increasing its volume (decreasing its pressure) or from fixture contact or the expanding gas medium. Increased stabilization time will allow these effects to become stable before the test data period begins. Test times are selected based on required leak rates or pressure decay criteria along with the package volume. See Annex A1 for further discussion.

9.3 For porous packages, it is necessary to coat the porous material with a coating that transforms the porous material into a non-porous material, as defined in ANSI/AAMI/ISO 11607-1, Annex C. Doing this will allow the evaluation of the package's seals and integrity of the non-porous side of the package. The selection of the coating and its use must not penetrate completely through the porous web and potentially occlude any defects in the seal area. The user must verify/validate that the coating is acceptable for this application. Evidence of suitability could be edge (cross-sectional) photographs of the coated porous material or any other suitable method.[4]

10. Report (Test Methods A and B)

10.1 Report the following information:

10.1.1 Method used.

10.1.2 Package type, size, materials, and lot numbers should be traceable.

10.1.3 Whether the package was tested empty or filled with product.

10.1.4 The apparatus used and settings for test pressure, timers, and decay limits. Other optional apparatus settings may be recorded such as restraining plate gap.

10.1.5 Date, time, location, and operator's name.

10.1.6 Conditioning parameters and environmental conditions at the time of test (if applicable).

10.1.7 Package test number and pressure decay if pressure decay limit was exceeded.

11. Precision and Bias[5]

11.1 This interlaboratory study was conducted to evaluate the precision of the pressure decay test method of leak detection in identifying a known leak in various sealed, nonporous empty packages. Two variations of the test method were examined, with Test Method A allowing the pressurized packages to expand without restraint, and Test Method B utilizing rigid restraining plates to limit package expansion under pressurization. Each of five laboratories tested ten randomly drawn test specimens from each of three materials under each of the two test methods, A and B. Materials were chosen to represent a range of products for which the test methods are suitable. The design of the experiment was similar to that of Practice E691.

11.2 The precision information given as follows represents pressure decay as measured in psig. The terms "repeatability limit" and "reproducibility limit" are used in accordance with Practice E177.

Test Method A—No Restraining Plates

Material	Pressure Decay Average, psig	95 % Repeatability Limit (Within Laboratory)	95 % Reproducibility Limit (Between Laboratories)
Foil pouch	246.020×10^{-4}	99.655×10^{-4}	191.932×10^{-4}
Film pouch	217.200×10^{-4}	54.370×10^{-4}	67.169×10^{-4}
Foil tray	48.240×10^{-4}	21.723×10^{-4}	27.482×10^{-4}

Test Method B—With Restraining Plates

Material	Pressure Decay Average, psig	95 % Repeatability Limit (Within Laboratory)	95 % Reproducibility Limit (Between Laboratories)
Foil pouch	149.560×10^{-4}	32.283×10^{-4}	32.283×10^{-4}
Film pouch	195.540×10^{-4}	13.748×10^{-4}	14.918×10^{-4}
Foil tray	64.900×10^{-4}	19.629×10^{-4}	26.095×10^{-4}

11.3 The standard deviations among test results are as follows. These standard deviations are multiplied by a factor of 2.8 to yield the respective limits previously stated.

Test Method A—No Restraining Plates

Material	Repeatability Standard Deviation	Reproducibility Standard Deviation
Foil pouch	35.591×10^{-4}	68.547×10^{-4}
Film pouch	19.418×10^{-4}	23.989×10^{-4}
Foil tray	7.758×10^{-4}	9.815×10^{-4}

Test Method B—With Restraining Plates

Material	Repeatability Standard Deviation	Reproducibility Standard Deviation
Foil pouch	11.529×10^{-4}	11.529×10^{-4}
Film pouch	4.910×10^{-4}	5.328×10^{-4}
Foil tray	7.010×10^{-4}	9.320×10^{-4}

NOTE 8—The study of these test methods was designed to define the precision of the test methods in cases where a leak is present. The previously stated precision data is based on an assumed "leak" in each package sample through a fixed orifice measuring 12.7 μm. This calibrated orifice was included in the internal tested volume of each package to simulate a "pinhole" leak. This artificial leak was programmed into the test design because a consistent actual leak of this size cannot be manufactured into the package.

11.4 Bias in the sense of a consistent difference in test results from an accepted reference value does not exist because the test results are defined by the test method itself. However, in order to standardize test conditions, an artificial bias was built into the test design in the form of a controlled leak, which

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1024.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1016.

was intended to define the universe of possible test samples as packages with identically sized leaks.

NOTE 9—The materials used in the evaluation of these test methods were chosen to represent a range of applications suitable for the Pressure Decay Leak Test. The test results for each material are uniquely related to its geometry and manufacturing characteristics. Because foil tends to bend and crease under pressurization, the foil pouches in the unrestrained test inflated into slightly variable shapes and volumes, which may have resulted in less repeatable pressure decay test results. The film pouches, though inflating to a consistently smooth surface, were subject to stretching when unrestrained that again may have yielded less precision. The test designer may wish to consider the use of restraining plates when testing these or similar materials in order to maximize the precision of the Pressure Decay Test Method.

12. Keywords

12.1 flexible packaging; leak test; medical packaging; pressure decay testing; restraining plate test; ISO-11607

ANNEX

(Mandatory Information)

A1. A GUIDE TO DETERMINING TEST PARAMETERS

A1.1 Several parameters are indicated in the test methods that are important to establish for the particular materials and package being tested so that an effective leak rate can be found from the pressure decay output of the test. The following information is a guide to determining the relationship of pressure decay and leak rate.

A1.1.1 From the ideal gas law $PV = nRT$, we can see that pressure, volume, and temperature are variables. Assume for now that temperature is constant.

A1.1.2 Using the ideal gas law and introducing time to establish a rate of change of gas volume lost to leakage, a relationship of changes in pressure in the system volume can be derived. This derivation will not be shown here. However, the relationship reduces to a relatively simple equation as follows:

$$Q\ (sccs) = \frac{\Delta P(atm) \times V\ (cm^3)}{\Delta t\ (s)} \tag{A1.1}$$

where:

Q = leak rate,
ΔP = pressure change measured in the package, and
V = initial volume of the package plus system volume, which is then divided by Δt, the time elapsed during the test readings of pressure.

A1.1.3 As an example, for a 100-cm^3 package measured for 30 s, with 0.001 psi (6.8×10^{-5} atm) as a pressure change, the resultant leak rate would be 2.26×10^{-4} sccs. Using this example, several implications for establishing the test parameters become clear. However, the establishment of a specified leak rate on a particular package and the leak rate relationship to use for maintaining sterility, preventing moisture intrusion, or other requirements is strictly up to the person setting the specification.

A1.1.4 *Package Volume Considerations*—From the leak equation, it is apparent that leak rate is directly related to total volume of the system being measured. The system volume consists of the package internal dead volume (the volume of the container less any internal solid product) and any volume of connection tubes, fittings, and instrument internal volume. This relationship enters into the decision on the appropriate use of restraining plates in certain circumstances, as restraining plates reduce the internal volume of the pressurized package (see Appendix X1 for further discussion of the use of restraining plates).

A1.1.5 *Equipment Sensitivity Considerations*—Another ramification of the leak equation is that as the resolution of the leak test measuring instrument increases, that is, the lower the pressure change it can detect, the rate of leak that can be detected decreases. Thus, in theory, the instrument with the highest pressure decay resolution will provide the most sensitive leak test. However, in the decision-making process, this conclusion must be hedged with the practical issue that highly sensitive instruments are more inclined to be affected by temperature effects and the electronic stability of the instrument readings can be problematic.

A1.1.6 *Test Time Considerations*—The time allowed for measurement will affect the leak rate inversely. If the time allowed for the test is increased, then the leak rate will decrease, thus decreasing the leak rate capability (increasing sensitivity). Leak rate sensitivity in a pressure decay test is then ultimately "only a matter of time"; even large volumes on low-sensitivity equipment can theoretically achieve low leak rates given a long enough test time. However, most production environments require short test times for efficiency. In addition, instrument limitations relating to temperature and electronic stability again become practical issues with long test times. If the reduction of test time without decreasing the sensitivity of the test is an important issue for the user the use of restraining plates may be worth investigating because of the plate's effect in reducing the effective internal package volume.

APPENDIX

(Nonmandatory Information)

X1. DISCUSSION ON THE APPROPRIATE USE OF RESTRAINING PLATES IN THE PRESSURE DECAY LEAK TEST METHOD ON FLEXIBLE, NON-POROUS PACKAGES

X1.1 Close examination of the precision estimates for the Pressure Decay Leak Test Methods on Flexible, Nonporous Packages shows a clear and statistically significant difference in the repeatability of pressure decay measurements between Test Method A (unrestrained) and Test Method B (using restraining plates). This increased precision in the restrained testing is particularly apparent in the foil and film pouches which are less rigid and, therefore, more deformable and expandable than the foil-sealed trays. There are several characteristics of testing with restraining plates that can contribute to this result.

X1.2 The use of restraining plates as accessories to the Pressure Decay Leak Test Method on flexible, nonporous packages accomplishes two purposes. One purpose is to limit the tested internal volume of the package, which is desirable under some circumstances to increase the sensitivity of the test method. For example, when a 10.0 by 15.0-cm (4 by 6-in.) package is pressurized, it can have a volume of over 500 cm^3 but when the same package is placed in a restraining fixture with the plates at a separation of 0.63 cm (0.25 in.), pressurization will yield a volume of approximately 100 cm^3. Because fill time for pressurizing a smaller volume will be shorter and because pressure decay is a function of internal package volume and time, reducing the internal volume of a package with restraining plates may reduce the time needed to accomplish the pressure decay test method.

X1.3 The second purpose that may be accomplished by the use of restraining plates is to increase the seal strength pressure capacity of the package, which allows the package to be tested at a higher pressure without fear of peeling or bursting the seals. For example, a peelable seal flexible package which has a burst seal strength of 6.9 kPa (1 psi) when unrestrained may have a burst seal strength of 69 kPa (10 psi) when restrained. An effect of increasing the test pressure is to reduce the test time necessary to detect a desired leak rate. This effect should be examined when appropriate to the package to be tested.

X1.4 Pressure decay test methods are guided by the combined gas laws $P_1V_1/T_1 - P_2V_2/T_2$ and the ideal gas law $PV - nRT$ (where P is pressure, V is volume, T is temperature, n is moles, and R is the universal gas constant). Because pressure decay test methods require a fixed or stable volume to measure, the elastic nature of the package walls can affect the sensitivity of the test method if stability of the package walls cannot be obtained. Most common package films, even those whose walls are difficult to stabilize under pressure, can be stabilized in restraining plates at the typically low pressures of these test methods.

X1.5 In order to prevent blocking of pinhole leaks in the walls during the pressure decay test methods, the surface of the restraining plates should provide limited porosity. Several techniques have been used to provide a means to prevent blocking or lowering of leak rate in package material walls in contact with the plates, including use of semi-porous plastic, scoring of plate surfaces, and use of screen-type materials. This factor should be kept in mind when considering the use of restraining plates, and the user should establish by experimentation any limiting effect on the pressure decay (leak rate) sensitivity.

X1.6 Note that the use of pressurized packages in restraining plates creates large forces applied to the plates and their fastening screws or devices. Caution must be used to prevent failure of the plates or fasteners, and only restraining fixtures from a qualified design source using appropriate materials should be used. Always use appropriate shielding and safety equipment.