Leak, Flow and Package Testing 101
Part 3. Non-destructive Pressure/Vacuum Decay Chamber Testing for Sealed Products or Packages

Part 3 of a 3 part paper designed to help you better understand why leak, flow and package testing is important. This series will enable you to make informed decisions as to the type and method of testing best suited to solving your issue.
Presented by TM Electronics, Inc.

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What's covered in Part 3

Part 1: Device / Product Integrity (Download separately)

Part 2: Package Testing (Download separately)

Part 3: Non-Destructive Pressure/Vacuum Decay Chamber Testing for Sealed Products or Packages

Surrogate chamber testing using pressure or vacuum decay

In the course of looking at leak and package testing methods, we have looked briefly at several non-destructive test methods, including:

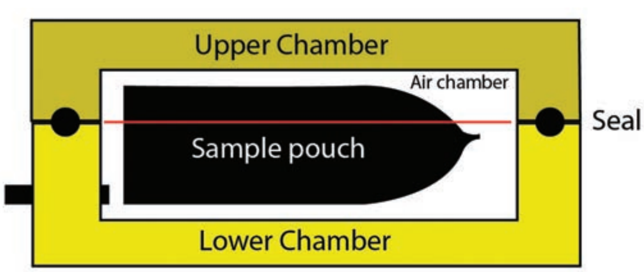
- Force Decay
- Displacement Decay
- Trace Gas Detection
- Helium Mass Spectrometry
- Pressure/Vacuum Decay Chamber Testing

How does chamber testing work?
The chamber testing method permits the non-destructive leak testing of sealed parts or non-porous packages by placing them in a chamber, then pressurizing (or evacuating) the chamber to the required test pressure and measuring the pressure or vacuum decay inside the chamber.

Air entering the part through a leak (or in the case of a vacuum test, leaving the part through a leak) provides the measurement of leakage into the test part.

What can be detected
In addition to leak testing closed or sealed products, this non-destructive method of testing will detect leaks from pinholes, cracks, seal and channel leaks in the walls or seals of common package materials such as films, foils and laminates. Packages can be preformed lidded trays, blister packs, or pouches.

Introduction to Surrogate Chamber Testing Using Pressure/Vacuum Decay



Why create a surrogate chamber?
Traditional pressure decay testing supposes a test item or package that can be pressurized. If your product or package is closed or sealed so it cannot be pressurized from an external source, an alternative and non-destructive method of pressure decay leak testing involves creating a closed space around the test item (a surrogate chamber) and pressurizing (or evacuating) it. A pressure differential can thus be created across the non-porous product or package walls or seal. Once stabilized, air movement from the higher pressure to the lower will indicate the presence of a leak path, providing a quantitative measure of package or product leak integrity without disrupting the package seals.

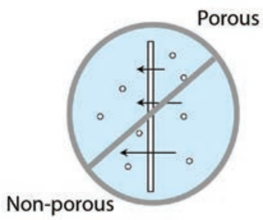
Volume around test object must be adequate
Because air moves in or out of the package or closed product in the presence of a leak, the air volume around the test object must be adequate to create a detectable change in the chamber pressure.

Keep in mind, when you minimize the interstitial volume (the area around the package, which will be subject to pressure or vacuum during the test) and maximize the instrument resolution (within reason), about 10^{-4} standard cubic centimeters per second (sccs) is an achievable sensitivity.
The method is quantitative; your test results are amenable to SQC (control chart) analysis process control.

Understanding your test item

1. What materials comprise your test item or package?
Materials (package walls and seals, closed product surfaces etc.) must be non-porous to air movement. Examples of packaging materials suitable for chamber testing are:

- Film/Film
- Foil or Laminate
- Lidded Thermoform
- Foil Sealed Bottles



Paper and Tyvek® are not suitable for this type of testing, as they are porous to air movement. Examples of closed products suitable for chamber testing include closed ended extrusions, vials, bottles, and welded housings.

2. If you are testing a package, KNOW YOUR SEAL STRENGTH.

When leak testing a package, the leak test pressure cannot approach the burst seal strength. TME engineers recommend that the leak test pressure not exceed 25% of the package burst seal strength. Seal strength can be quantitatively determined by using burst, creep and creep-to-failure testing.

Stresses applied
These tests require pressurizing the entire package and measuring the peak rupture pressure (burst test). These inflation tests provide three different components of stress to the package: peel stress with horizontal and vertical components, tension due to hoop stress in the vertical direction, and lateral stress due to package expansion. If these stresses are greater than the strength of the seal at any point within the package, the seal will rupture. Inflation testing provides a whole-package minimum seal strength and also indicates the weakest seal area, and is equally applicable to peelable and non-peelable seals.

What can be tested this way
Inflation tests are applicable to most package forms such as pouches, header bags, lidded trays, flexible or rigid blisters and laminated or rolled tubes. Keep in mind that inflation seal strength testing is destructive.

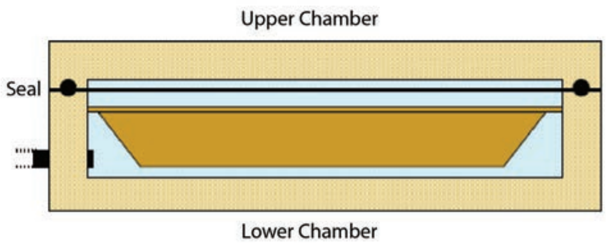
An illustration of seal test

Establishing your seal strength

TME's BT Integra-Pack is an intelligent package test system for burst, creep, creep-to-failure and leak integrity testing.

The BT Integra-Pack features a number of test modes for conducting testing on both nonporous and porous packages without having to change the setup, test item or instrument settings.

Designing your surrogate chamber

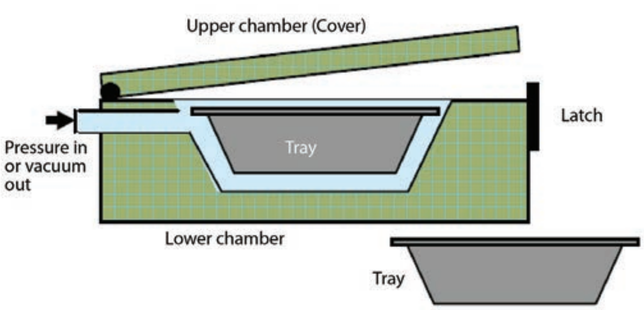


The chamber must be sealed from the atmosphere. This creates the vacant space that will be pressurized or vacated to perform the test. Care must be taken that the seal is strong enough to prevent air leakage when pressurized or vacated. When the test pressure in the chamber space has been stabilized, you will measure leakage by pressure change in vacant chamber space over a predetermined period of time as pressure leaks into (or out of, in case of vacuum test) the test item or package.

Minimize interstitial volume

It is also important to minimize the interstitial volume of the chamber (the vacant chamber space surrounding the test item or package). The smaller the interstitial volume, the more sensitive the test.

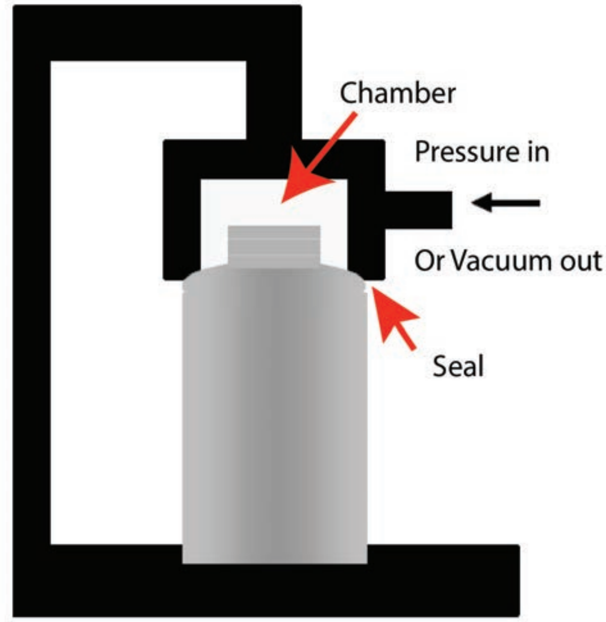
Examples of how chambers work



Typical chamber fixture to accommodate pre-formed, filled and lidded trays

How it Works

The tray is inserted into the test chamber and the cover is locked down. The airspace in the chamber is then pressurized (or evacuated), stabilized and tested for pressure (vacuum) decay. No decay, no leaks; if the seal leaks, there will be measurable pressure or vacuum decay.



Fixture for non-destructive testing of induction welded bottle seals

How it Works

The fixture head is lowered onto the bottle shoulder where a seal is made. The airspace in the chamber thus created is pressurized (or evacuated), stabilized and tested for pressure (vacuum) decay. No decay, no leaks; if the induction welded seal leaks, there will be measurable pressure (vacuum) decay.

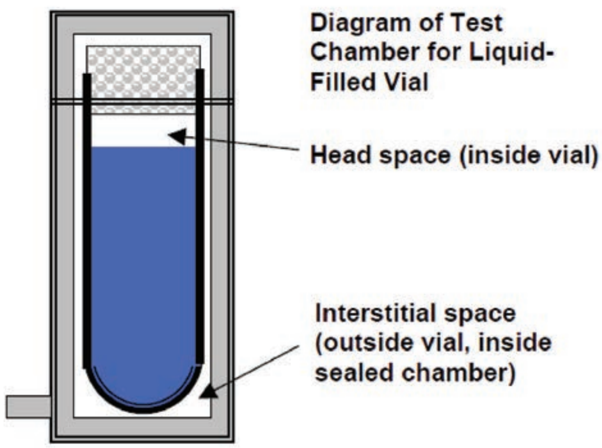
In both of these examples, we see how the interstitial space has been minimized by designing the fixtures specifically around the shape of the test item.

Caveat: Adequate headspace is necessary

The nature of the pressure or vacuum decay test requires a minimum head space inside the closed product or package. "Head space" refers to the amount of air enclosed in the test item or surrounding the product inside a package.

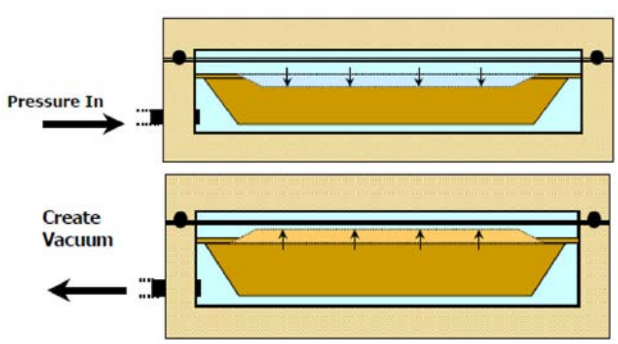
Detection of interstitial space

High resolution instrumentation can detect pressure changes as small as 0.0001 psi in the interstitial space (the space surrounding the closed product or package). In order to detect this pressure change in the interstitial space, there must be sufficient air in the head space relative to the air in the interstitial space to create this much of a change in pressure.



When should Vacuum Decay Testing be used?

The use of pressure or vacuum for your chamber test is related to the structural rigidity of your package or product, since pressurizing the interstitial space (the space between your test sample and the chamber walls) may damage a fragile object or the contents of a non-porous pouch. A good example is a potato chip bag. Depending on the amount of head space inside the package, pressure decay testing could result in broken chips.



Pressure decay testing is generally the method of choice, because the nature of the pressure decay test allows greater consistency and repeatability in your test results.

Why is Pressure Decay the preferred method? The interstitial volume of the chamber (the air space inside the chamber surrounding the package or product) is consistent in size from test to test when pressurized. In a vacuum test, however, the air in the package or product head space (the air space inside the package or product) will cause expansion of the walls of the test object.

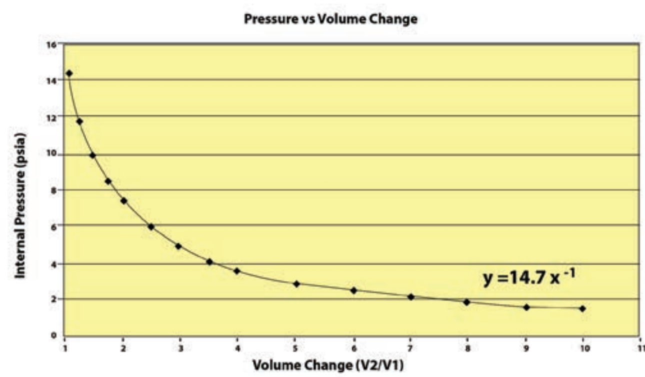
Impact of flexible materials

With flexible materials such as pouch walls, the head space volume may vary from sample to sample, which would lead to varying expansion in the presence of a vacuum in the interstitial space.

The basic gas laws define this package internal pressure effect;

$$P1\ V1 = P2\ V2$$

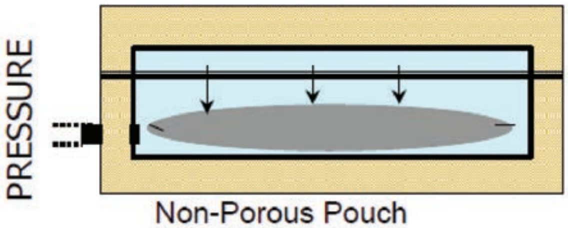
Which we can see in the graph below.

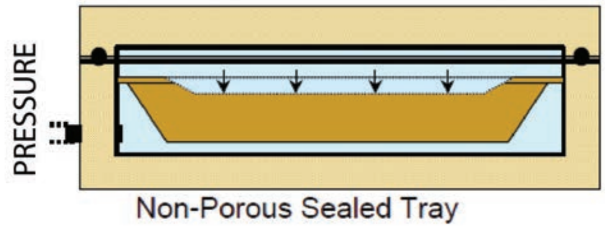


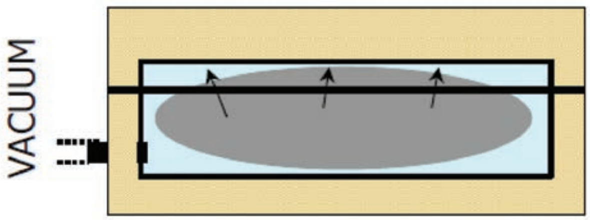
According to this, we see how pressure and volume are related. Therefore, if there is variation in the expansion of test items in a consistent vacuum, then it follows that the volume of the interstitial space, where we are measuring vacuum decay, would vary from test to test. This would make it very difficult to achieve repeatability in test results.

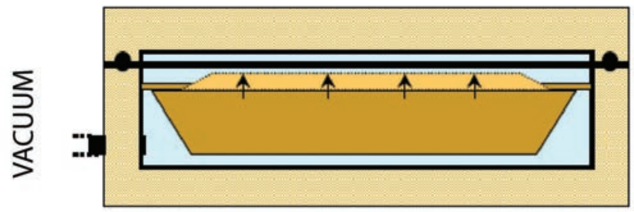
Test sensitivity

A related issue involves the sensitivity of the test. In the vacuum test, if the flexible sample walls stretch under vacuum, the pressure inside the test item drops (again, the relationship between pressure and vacuum). This drop in internal pressure decreases the differential pressure you are trying to detect, which impacts the sensitivity of the vacuum decay test.









Other influences on test sensitivity

We have already mentioned the fact that minimizing the interstitial space produces the maximum sensitivity in your test.

If you recall the Leak Rate equation (see Part 1 which can be download) and substitute the interstitial volume VC for the system volume V, you can see the chamber leak effect:

$$Q = \Delta P * Vc / \Delta t$$

where Q is the leak rate, **Δ**P is the change in pressure in the chamber after stabilization, and **Δ**t is the test time. Because pressure decay is a volume function, minimizing the chamber volume maximizes the sensitivity of the test.

FYI: The sensitivity of your pressure/vacuum decay chamber test is also dependent on the sensitivity of the instrument used to detect the change in pressure. The TME Solution-C Chamber Leak Test Instrument has a decay resolution of 0.0001 psi (0.01 mbar/sec), which when combined with a sealed chamber fixture can detect holes as small as 5 microns in diameter.

Chamber Testing Systems

TME Solution-C™ Chamber Leak Tester

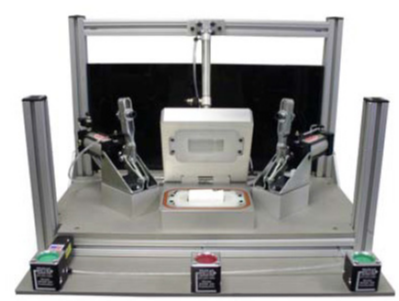
The TME SOLUTION-C test system produces quantitative test results from products that cannot be accessed to pressurize through an access port, as well as sealed, flexible medical, pharmaceutical and food packages. By combining the sensitivity of pressure or vacuum decay leak testing with the simplicity of sealed fixtures, the TME SOLUTION-C system can detect holes as small as 5 microns.

This highly sensitive method uses a proprietary chamber design to find leaks in product seals or walls and seals of common package materials such as films, foils and laminates widely used in industry today.

TME Solution-C induction welded bottle seal test system

The TME SOLUTION-C Bottle Seal Tester produces quantitative results in pharmaceutical, nutraceutical or food bottles by combining the sensitivity of pressure decay leak testing with the simplicity of sealed fixtures.

This highly sensitive method uses a proprietary chamber design to find leaks in walls or seals of common bottle sizes widely used in industry today, including those with various shapes and neck or screw sizes. Fixtures are custom designed to accommodate the bottle under test; upright fixtures can test bottles containing liquids (with head space) and horizontal fixtures can be manual or semi-automated



TME Solution-C sealed package system

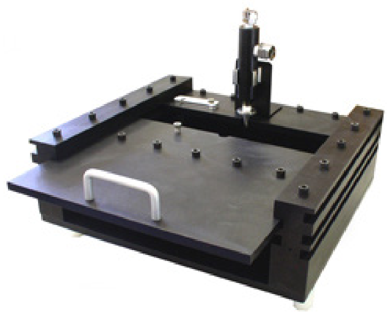
In-process leak testing will detect leaks from pinholes, cracks, seal and channel leaks without disrupting your production process.

The TME Solution-C System produces quantitative results in flexible medical, pharmaceutical and food packages by combining the sensitivity of pressure decay leak testing with the simplicity of sealed fixtures.

Packages can be pre-formed lidded trays, blister packs, or pouches. Holes as small as 5 to10 microns can be detected in package walls. The TME Solution-C Non-Destructive Chamber Test System can be custom designed to suit your individual product and testing requirements.

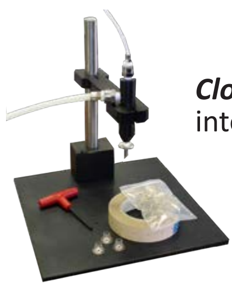
Examples of standard and custom test fixtures

TME is know for its ability to design and manufacture custom test fixture design to fit your unique testing needs. TME has designed over 1,000 customer fixtures .



Restraining plate fixture - stabilizes expansion during pressure decay testing

Open package test fixture - for flexible package seal strength measurements

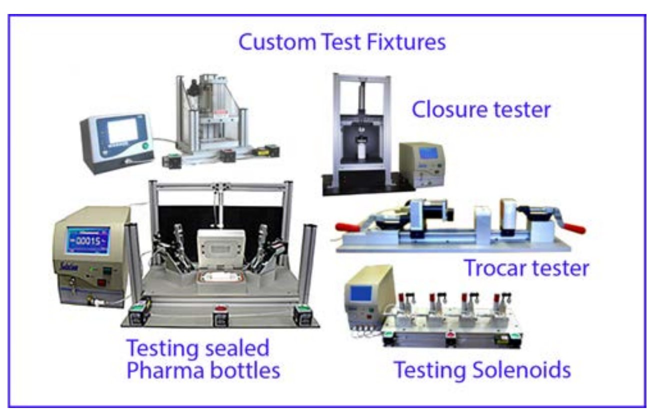


Closed package test fixture - for closed package seal strength and leak integrity measurements - includes probe assembly and package access kit

Open package test fixture - for large flexible package seal strength - up to 24 inches

Radial sealing test fixture - for testing tubular products

Just a small example of custom test fixtures



Quiz

Test your understanding of the pages part 3.

Q.1. Non-Destructive testing methods are?
- A. Force decay
- B. Displacement decay
- C. Trace gas detection
- D. Helium mas spectrometry
- E. Pressure/Vacuum decay chamber testing
- F All of the above

Q.2. Pressure /Vacuum decay chamber testing:
- A. Is destructive
- B. Is non-destructive

Q.3. Items that can be tested by this method are
- A. Porous packages
- B. Non-porous packages
- C. Sealed parts
- D. B and C
- E. A and C

Q.4. This method is only good for testing for gross leaks?
- A. True
- B. False

Q.5. Decay testing can only be used if the item or package can be pressurized?
- A. True
- B. False

Q.6. Interstitial space is
- A. The space in the Interstal
- B. The space in a chamber not taken up by the item being tested
- C. The space between the Troposphere and the Mesosphere

Q.7. Sensitivity of a chamber test is not impacted by the volume of the interstitial space?
- A. True
- B. False

Q, 8. Paper and products that function similar to Tyvek® are?
- A. Excellent products to test by this method
- B. Only good for writing on
- C. Are porous and not suitable

Q,9. Determining seal strength is important because?
- A. You want the package to rupture before the seal
- B. You want the seal to leak before the package ruptures
- C. The seal with the most strength is usually leader of the colony

Q,10. Factors to consider when designing a surrogate chamber are:
- A. The chamber must seal out the atmosphere
- B. The seal must be strong enough to prevent leakage when pressurized or vacated
- C. The volume of the interstitial space
- D. All of the above

Q, 11. Headspace is?
- A. Volume of air inside the product or package
- B. Interstitial volume
- C. Distance above the test fixture
- D. A and B

Q, 12. Volume of headspace is important because there must be sufficient volume to create a change of pressure
- A. True
- B. False

Answers:
Q1. F. All of the above. Q2. B. Non-destructive Q3. D. Non-porous packages and sealed parts. Q4. B. False. Method will detect leaks from pinholes, cracks, seal and channel leaks.Q5. False. A surrogate chamber enables a pressure differential to be created across the product or package walls or seal. Q6. B. Q7. False. The smaller the interstitial volume, the more sensitive the test. Q.8. C. They are porous Q9, B, Its a seal test. Q10, D. All of the above and more. Q11, D. A and B. Q12, A. True.
Tyvek is a registered trademark of DuPont

Glossary of Terminology

Pressure Decay Test: an inflation leak test in which a non-porous package or product is pressurized to a preset level. After the package has stabilized, the decay in pressure over a preset test time is evaluated to determine if a leak is present.

Vacuum Decay Test: similar to the Pressure Decay Test, except that a preset vacuum is established inside the product or package, and the decay in the vacuum over a preset time is evaluated to determine if a leak is present.

Decay: refers to the change of pressure (Δ P) inside a pressurized containment during a pressure decay leak test. Decay can refer to either positive or negative (vacuum) pressure

Pressure/Vacuum Decay Test Cycle:
Consists of five consecutive steps:
1. Load (attach the test item to the test system)
2. Charge (pressurize the test item to a preset pressure, or create a predetermined vacuum level)
3. Settle (time allowed for the volume of the test item to stabilize to minimize the effects of material stretching, adiabatic heating, etc.)
4. Test (the time during which the decay in the pressure or vacuum is measured
5. Unload (removal of the test part from the test system).

Decay Curve: In a pressure decay leak test, the graph of the drop in pressure (Y axis) over time (x axis) is called the decay curve. TME uses the decay curve in its “Test Plot” graphic display and in its “Memory Reference Curve” technology, in which the decay curve for an acceptable test part is determined and reject decisions are made by the test instrument by comparing the test decay curve to the acceptable “memory reference curve” for the test part.

Resolution vs. Sensitivity:
Resolution is the least significant digit that an instrument is capable of measuring; for example, the TME Solution Leak Tester has a resolution of 0.0001 psi.
Sensitivity is the smallest volume leak rate your test system (including the air lines, fixtures, etc.) can detect.

Units of Measure:
Pressure units of measure include: psig (pounds per square inch gauge), Pascals, kg/cm2 and many others.

Flow rate units of measure include: liters/min, sccm (standard cubic centimeter per minute), sccs (standard cubic centimeters per second) – where standard refers to atmospheric pressure.

Transducer: Any sensor that converts a physical parameter (for example, pressure) into an electronic signal that can be utilized by an instrument.

Leak Rates:
Volume Leak Rate: change in volume per unit of time (measured in Flow Rate units of measure, see above)
Pressure Leak Rate: change in pressure per unit of time (measured in Pressure units of measure, see above)

Operating Test Parameters: the descriptive factors defining a leak, flow or package test. These may include:
Charge, settle, and test times for pressure or vacuum decay tests;
Test pressure;
Flow rate into the test item (very important in burst testing);
Maximum acceptable volume leak rate.

Sequential vs. Concurrent Testing:
Concurrent testing enables leak tests to be performed simultaneously on more than one and up to four test items in a Solution tester, with one test item connected to each port on the instrument. The tests must have identical test parameters (test time, pressure, decision point etc.), and the test results are discrete and identifiable to a specific test part. An instrument of this type has individual transducers for each test port.

Sequential testing involves performing a series of like tests on a test item through a single port. An example is a leak test followed by a flow occlusion test on a test item and/ or a series of leak and flow tests on multiple ports. An instrument of this type may have one port or multiple ports that are tested one at a time.

Occlusion Testing: An occlusion is a partial blockage of a flow path. An example is a crimp in a catheter. Occlusion testing can be done in several ways, including:
1. mass flow rate
2. back pressure measurement
3. pressure drop measurement.

Back pressure: the pressure forcing air through a leak path.

Package Testing: Based on international standards and FDA guidelines, thorough package testing should consist of both seal strength testing and leak integrity testing.

Seal Strength Testing: a destructive test that provides a measurement of the strength of a package seal of a porous or non-porous package. Seal strength testing can also identify the area of weakest seal. Seal strength testing can be done using inflation tests or tensile tests, but TME recommends using one or more of the following inflation seal strength tests:

1. Burst testing (recording the peak or ultimate strength of a package seal);
2. Creep Testing (measuring resistance to seal peel) – result is pass/fail only;
3. Creep-to-Failure (measuring resistance to seal peel) – result is variable statistic (time).

Integrity Testing: a measure of the quality of the package or product in general, including the seal areas and the package or product materials themselves. Leak Integrity Testing generally refers to product or package leakage measured by a leak test.

Fixtures: Fixtures are used to enable the test instruments to perform specified leak, flow or package tests on a variety of products or packages. Examples of fixtures commonly used by TME include: *Open Package Test Fixtures, Closed Package Test Fixtures, Restraining Plate Fixtures, Package Probe Assembly, Radial Sealing Fixtures.* Fixtures are often custom designed to accommodate a customer's very specific testing need.

Closed Package Entry System: a method to obtain a leak tight measuring path between the package interior volume and the instrument's pressure transducer. TME uses the patented Package-Port System, which consists of the following disposable items:

1. Package-Port – a reusable plastic entry port which accommodates the pressurizing sensor probe, and
2. Adhesive Disks that adhere the Package-Port to the surface of the test item which are supplied in rolls of 1000.

Non-Destructive (Chamber) Testing: a method to non-destructively test a sealed, non-porous package or product for leaks. It is necessary that the test item contain some air or other gas inside – this is called the "head space". The package or product is enclosed in a surrogate chamber that provides an interstitial air space around the test item. This air space is then pressurized and stabilized, and decay of the pressure in this air space (indicating air leaking into the head space of the package or product) is measured. A chamber test can also be done using vacuum.

Surrogate Chamber: the test chamber used in non-destructive chamber leak testing is called a "surrogate chamber" because the actual pressure or vacuum decay leak test is done on the air contents of the chamber surrounding the test item rather than on the test item itself.

NEMA-4: A designation in the USA which indicates that an item (such as case, components, or an assembly) can withstand damage from harsh industrial environments, including water or dust. The NEMA-4 designation corresponds to the IP-65 designation.

Verification/Qualification/Validation: These terms describe a process that is helpful when evaluating a new product or package manufacturing process:

1. Verification refers to the test and inspection results for each individual component and/or step involved in the manufacture and packaging of a medical device.
2. Qualification is a combination of verifications to determine how well equipment, materials, and a process can work together.
3. Validation is the combination of various qualifications and other objective evidence that the processes consistently produce prod uct meeting predetermined specifications.

IQ/OQ/PQ: Installation Qualification, Operation Qualification, Performance Qualification. These protocols are part of the validation process addressed above.

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