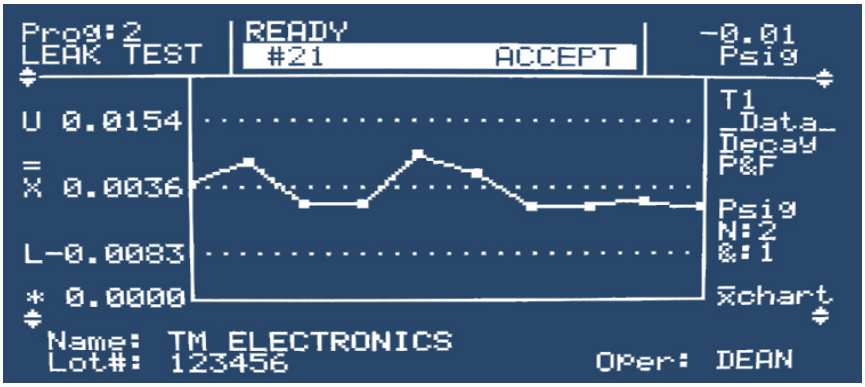
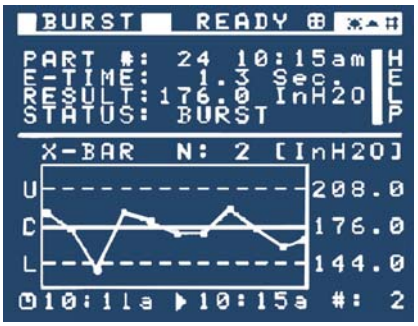
# Leak, Flow and Package Testing 101









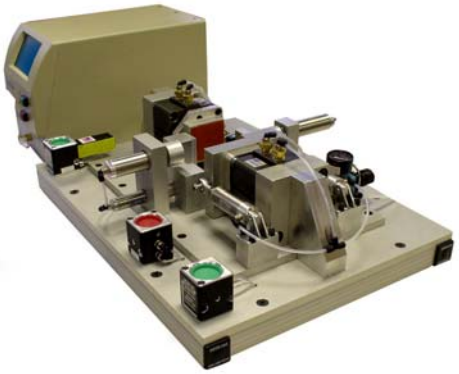
Presented by
TM Electronics, Inc.

**www.tmelectronics.com**

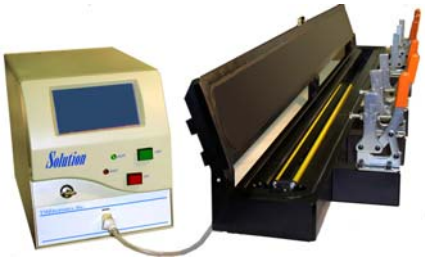
# *Why is Testing important?*


- Although Leak and Package Testing are different in their functional approach, the fundamental motivation for testing is the same - to insure that material that is supposed to remain in a package or product stays there, and that nothing in the outer environment that is not intended to get into the package or product can enter.


- You are taking this course because you are interested in basic leak, flow or package testing. Perhaps your product is designed to contain a material without losing any of the contents or to transfer a material or solution intact from one point to another. Perhaps you are introducing a new product that is itself enclosed in a completely sealed package, sterile or otherwise, to protect the product from the world, or to protect the world from your product.



- Whatever issue you are facing, it has become apparent that testing is important. Leaks mean *product failure.* Seal or closure weakness may lead to leaks. A leak or seal weakness may lead to material leakage, environmental contamination, loss of sterility or component failure. In all cases, leaks mean *waste of manufactured product,* and leaks that are not found will surely lead to *customer complaints!*





# *Course Syllabus*

## *Term One: Device / Product Integrity Testing*

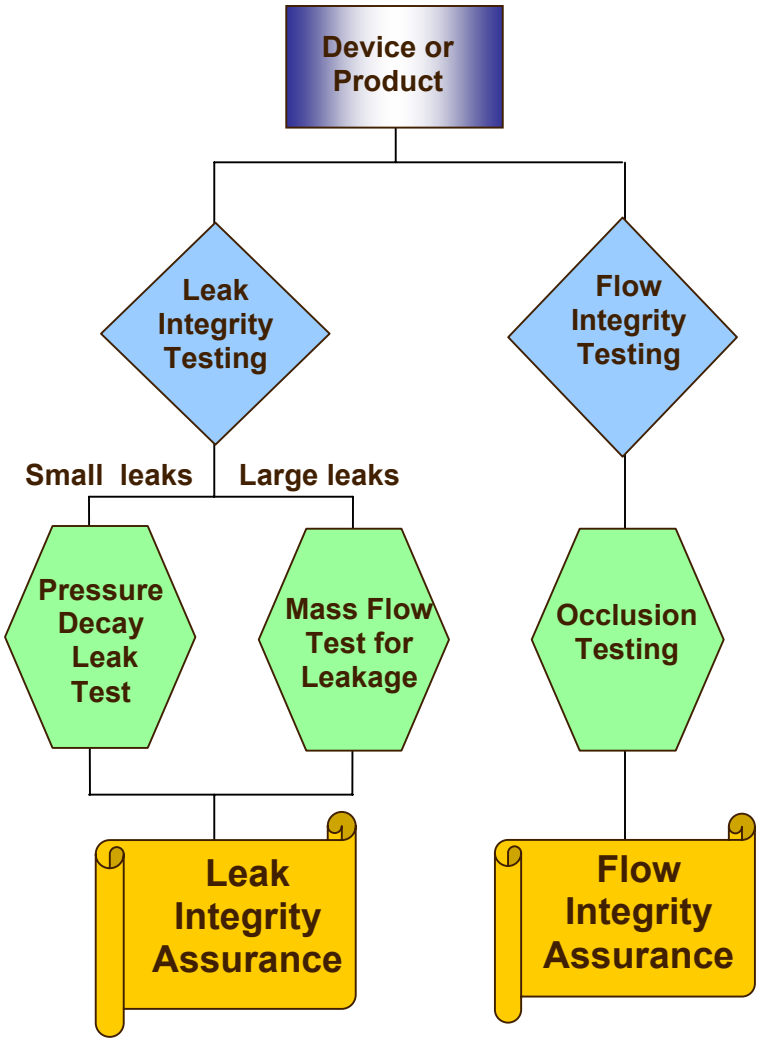


## *Term Two: Package Testing*



## *Term Three: Non-Destructive Pressure/Vacuum Decay Chamber Testing for Sealed Products or Packages*

# *First Term Overview: Device or Product Integrity Testing*




**Whether you manufacture medical devices, auto parts, or other products, it is essential to provide assurance of product quality.**

Leak and flow testing are a valuable way to enable your QC department to provide **assurance of your product's integrity.**

Large or small leaks can be quantified using pressure decay and mass flow testing, and flow testing can also identify obstructions in flow-through parts.

The end result is that you, the manufacturer, will gain CONTROL over your manufacturing process, and QUALITY ASSURANCE in the field.

## *What is a Leak?*

In this section, we will discuss how leakage is measured and what is meant by "Leak Rate." We will begin to look at the issue of determining the appropriate standard or specification for your particular product, and discuss the practical issues involving leakage including typical product leakage ("leak rate"), leak rate conversions and the price of "no leaks." Although important and fundamental in leak science, most information in this section relates to the identification and measurement of leaks; package integrity testing will be discussed in detail later in the course.

## *Unit 1: What is a Leak?*

Simply and directly, a leak is a hole or a path through which the package contents may escape, or through which ambient materials from the environment may enter. There are holes in everything; the issue you face is to decide how large a hole must be to cause a failure, and where that hole is likely to be located. Your answer to these questions will help to determine what kind of leak testing is most appropriate for your product.

There are two common methods of locating leaks: bubble testing, and "sniffer" testing with trace gas. We'll address these later in the class. A very important issue is the definition of the leakage – or leak rate – that must be found to avoid product failure, and this definition will vary according to your product and its circumstances.

Remember that everything leaks, even if it is the permeation of gas molecules through a metal or plastic, or atoms leaking through a lead shield. It's just a matter of time. The important point is that leakage is *relative to a standard or specification.*

In order to define the specification or standard (how much leakage is too much leakage?), and indeed in order to measure leakage at all, we need to understand one basic relationship:

$$\textbf{Leakage (or Leak Rate)} = \frac{\Delta V}{\Delta t}$$

where V is the volume of the medium exiting or entering and t is the time period during which you are measuring the change in volume. Leak Rate is therefore the volume of material (air, fluid etc.) that escapes from a closed or sealed containment in a predetermined amount of time. You may see leak rate expressed in various units of measure, such as cc/min, cc/sec, or $ft^3$/hr. The units will generally reflect whether you are measuring a relatively high or low leak rate; for example, leakage of air from a medical fluid container will be typically in the range of $1 \times 10^{-3}$ cc/sec (quite small) but air leakage from a water pump may be in the moderately high range of 8-10 cc/min and still be considered an acceptable part based on its use and test specifications. We will discuss the setting of test specifications later in this course.

Note that some people will use a leak measure as a change in pressure over time (psi/sec, kPa/sec). These measures represent the result of the volume leakage rate for that specific application. However, unless the total volume of the part and measuring system are known, these measurements cannot be standardized against other instruments or measuring systems.

Using volumetric or mass measurements provides the best approach to defining leakage in a test system. Using the pressure drop method is most useful when a particular part can be identified as a "good" or "bad" part.

## *Typical Product Leak Rates*

Having developed a very rudimentary sense of the meaning of "Leak Rate", let us take a look at some practical issues involving actual products. The following table demonstrates typical leak rates for a variety of medical products.

## TYPICAL ALLOWABLE AIR LEAKAGE FOR MEDICAL APPLICATIONS

| APPLICATION | PRESSURE | LEAK RATE | CYCLE TIME |
|---|---|---|---|
| ***Catheters*** | 30 psig | <1 cc/min (1.6 x $10^{-2}$ cc/sec) | 1 – 2 sec |
| ***Balloon Catheters*** | 200 psig | 0.6 cc/min (1 x $10^{-2}$ cc/sec) | 10 – 15 sec |
| ***Blood Bags*** | 2 psig | 1 – 4 cc/min (1.6-6.4 x $10^{-2}$ cc/sec) | 4 – 10 sec |
| ***Syringes*** | 10 – 150 psig | 0.1-5 cc/min (0.2-8 x $10^{-2}$ cc/sec) | 3-10 sec |
| ***Insulin Tester Containers*** | | 1 x $10^{-4}$ cc/sec | |
| ***Medical Fluid Containers*** | | 1 x $10^{-3}$ cc/sec | |

You may note that some items with higher allowable leak rates, such as the syringes, have a relatively short test time. This is related in part to the degree of flexibility of the test part. A less flexible test item such as a syringe may require less time to "stabilize" before the actual leak test begins, whereas a more flexible item such as a blood bag may need a longer "settle" time. This issue will be discussed in greater detail later in the class.

Similarly, the table below illustrates typical product leakage in industrial applications. Note again that variations in test specifications vary depending on the type of part. Several have a relatively high level of leakage to reach the critical point, but others are lower – in particular, the brake cylinders; consequently, a longer test time is needed to detect leakage at the desired critical level (remember our formula?). Another reason for variations in test time is the size of the part being tested. Even though the critical level of leak to be measured is larger, the volume of the part is larger. Most of these parts are not particularly flexible, as were many of the medical devices, but even these metal parts can be affected by temperature changes in the gas, and still need some stabilization time**. In general, we can state that the lower the leakage rate, the longer the test time required.**

## TYPICAL ALLOWABLE INDUSTRIAL PRODUCT LEAK RATE

| APPLICATION | PRESSURE | LEAK RATE | CYCLE TIME |
|---|---|---|---|
| ***Water Pumps*** | 15 psig | 4 – 6 cc/min | 10 – 15 sec |
| ***Oil Pumps*** | 30 psig | 8 – 10 cc/min | 5 – 10 sec |
| ***Thermostats*** | 80 psig | 1 cc/min | 2 sec |
| ***Radiators*** | 15 - 40 psig | 3 – 6 cc/min | 15 – 30 sec |
| ***Brake Cylinders*** | 80 psig | 1 x $10^{-3}$ cc/sec | 30 sec |
| ***Hoses*** | 150 psig | 1 cc/min | 10 sec |
| ***Tube Sets*** | 15 psig | 2 cc/min | 5 sec |
| ***Faucets*** | 80 psig | 5 cc/min | 15 sec. |
| ***Fuel Injection Units*** | | 5 x $10^{-4}$ cc/sec | |
| ***Diesel Injection Units*** | | 1 x $10^{-2}$ cc/sec | |
| ***Gas Filters*** | | 3 x $10^{-3}$ cc/sec | |
| ***Diesel Filters*** | | 3 x $10^{-2}$ cc/sec | |
| ***Gas Pressure Regulators*** | | 3 x $10^{-2}$ cc/sec | |
| ***Gas Tubes*** | | 3 x $10^{-3}$ cc/sec + high pressure test | |

## *Further Discussion: Leak Rate through an Orifice*

*Leak Rate* through an orifice – a hole or a break – is a function of several variables: the pressure differential across the orifice; the diameter or size of the hole; the density of the test medium; the temperature.

The relationships can be defined as follows:

$$Q = k * d^2 * \text{Sqrt} ( P_1^2 - P_2^2 / \rho * T_a )$$

where Q is flow rate, d is the diameter of the orifice, $P_1$ and $P_2$ are the pressure on either side of the orifice, $\rho$ is the specific density of the medium, k is a dimensional constant and T is the temperature of the system.

To obtain consistent measurements of leak rate, the temperature must be constant. When dealing with gases, most measurements assume it is used in a state where it is considered incompressible.

***Obviously, you can relate leakage with hole size, but several empirical factors are tied up in "k". These factors come from geometry and fluid flow properties like the Reynolds number. Because of this, most leak rates are approximate, unless they are measured directly by mass flow.***

*As an aside, because matter can flow through an orifice in either direction, in general, leak rates can be assessed using either pressure or vacuum. This is a part-related issue, and in making this decision you need to consider several points: the function of the part, the structural integrity of the part, the degree of pressure change needed to find the leak, and whether the part will "outgas" in a vacuum, giving false readings.*

## *Leak Rate Conversions*

A caveat to keep in mind when considering your test specifications: there are several common units of measure. The following tables give leakage and pressure conversion charts that may be helpful to you; note that these are volumetric leak rates at "Standard Conditions" – stated 70 degrees Fahrenheit, 14.7 psia (1 atm).

### *Leakage & Pressure Conversions*

All leak rate units are at standard atmosphere conditions (70°F, 14.7 psia)

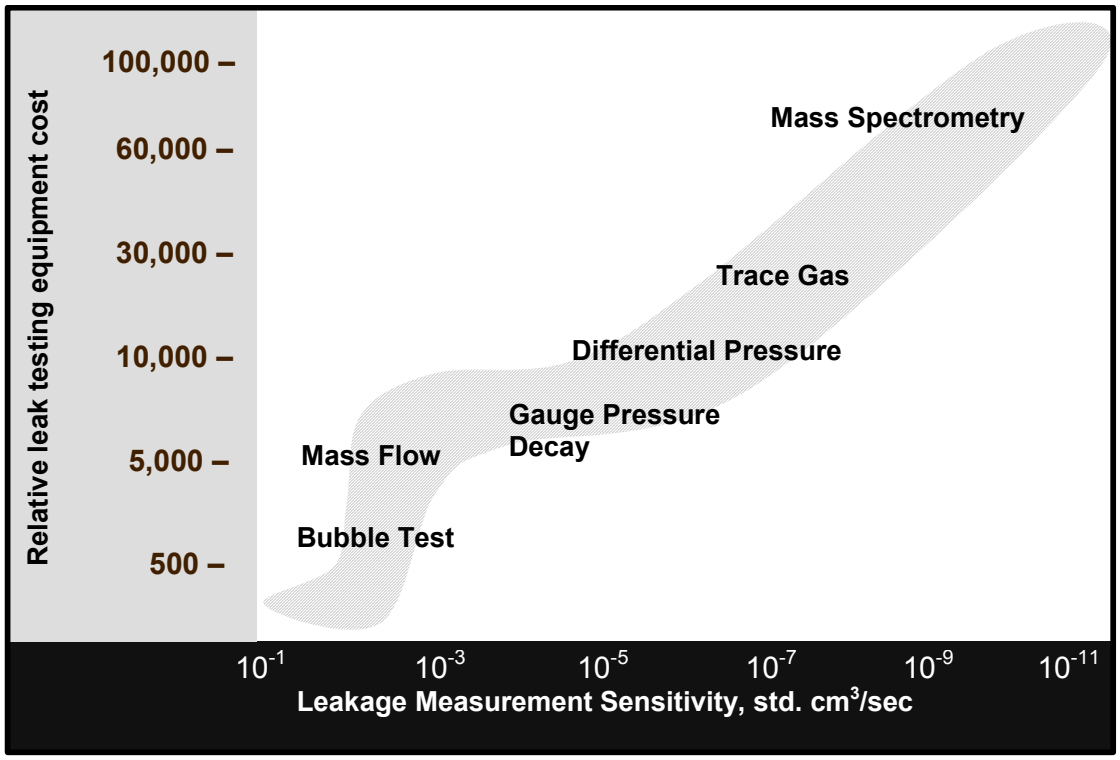
| *To Obtain* ➡ | | *cc/sec* | *cc/min* | *in³/min* | *ft³/hr* | *Pam³/sec* |
|---|---|---|---|---|---|---|
| *Multiply* | cc/sec | 1.000 | 60.0 | 3.66 | 0.127 | 10 |
| | cc/min | $1.67 \times 10^{-2}$ | 1.000 | $6.10 \times 10^{-2}$ | $2.12 \times 10^{-3}$ | 0.167 |
| | $in^3$/min | 0.273 | 16.39 | 1.000 | $3.47 \times 10^{-2}$ | 2.73 |
| | $ft^3$/hr | 7.87 | 471.9 | 28.80 | 1.000 | 78.7 |
| | $Pam^3$/sec | 0.10 | 6.0 | 0.366 | $1.27 \times 10^{-2}$ | 1.000 |

Be careful to note on your specifications – or on specifications from others – what "their" standard conditions are. Many will use 0 degrees Celsius as a reference. The SI units are $Pam^3/sec$ (P*V/t). Note also that the following are not leakage measurements (psi/sec, Pa/sec, mbar/sec), but are pressure rates. The actual leak rate is a function of volume. These pressure measurements are only good for one part design, at specific conditions.

## *The Price of "No Leaks"*

We have now developed a sense of the relationship between the size of a leak that is critical and the sensitivity of the test needed to find it. Once you have determined the size of the critical leak for your particular part or device, you can make a determination of your test specifications, and you can begin to research the test method to best serve your needs. It is important to realize during this specification-setting period that there is a cost associated with instituting this quality control step. The table below shows a relative scale of different test methods. There are wide bands around each method to accommodate the different instrument type and the fixturing required to implement the test for your particular part.

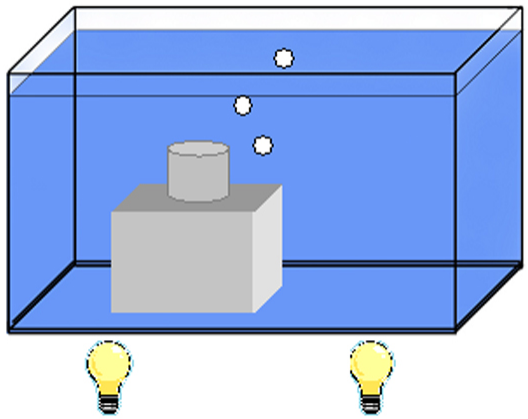
### *The Price of "No Leaks"*




As one would expect, the cost associated with greater sensitivity increases. If detection of very low leak rates is essential, the higher cost of equipment may be justified. Only you can analyze your own best interests.

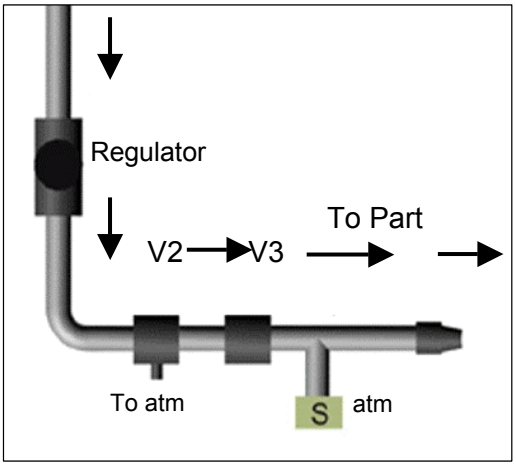
## *Unit 2: Types of Leak Tests*

### *Bubble ("Dunk") Testing*



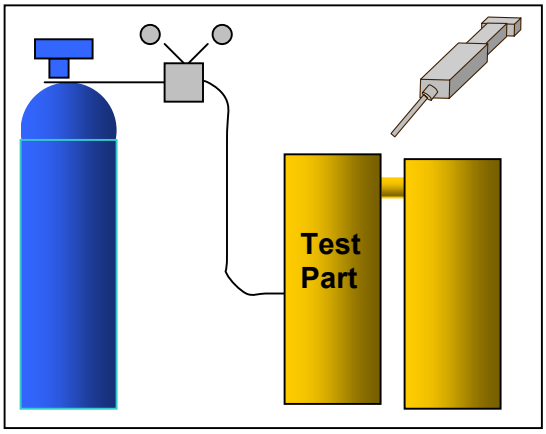
Bubble testing is the simplest, least expensive method of detecting and assessing leak rate. The procedure is as the name implies: the part being tested is submerged in water, and the test operator visually observes and takes note of any bubbles escaping from the test part. Under the best test conditions, including good lighting, a very clear liquid, and a patient, alert operator, a leak rate of $10^{-2}$ to $10^{-3}$ sccs can be observed (a 1-2mm bubble escaping). Disadvantages include a long test time (a minimum of 30 seconds per test), water contamination, and part clean-up time. The sensitivity of the test is not high, due to operator dependence.

### *Pressure Decay Testing*

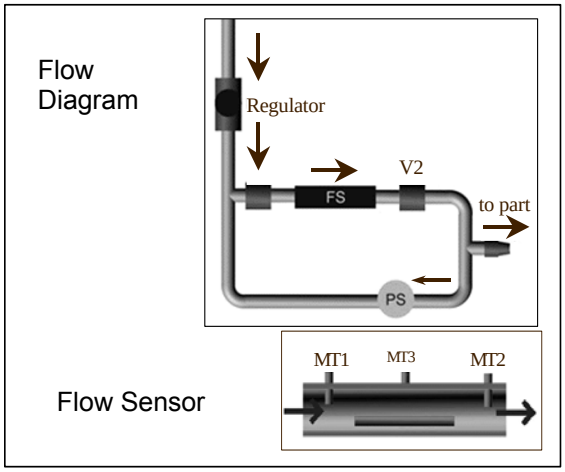



Pressure decay testing measures the change in pressure between atmospheric pressure and your pressurized test sample. Unlike the bubble test, this test method yields quantitative information, hard data points that can be recorded and upon which decisions can be made. This removes the dependency upon the operator and allows specific accept/reject criteria to be set, and this method is quite simple to use. It is reasonably fast; 2-4 second cycles are achievable, keeping in mind that test time is volume dependent. Although more sensitive than bubble testing, pressure decay testing is as sensitive as the time available for the test. A variation uses a differential transducer for pressure on both sides of the membrane, which might give more sensitivity in some cases but adds more complication (differential pressure testing requires a reference volume, and is temperature dependent on both volumes; it is difficult or impossible to "standardize"). Pressure decay may include vacuum testing since a "vacuum" is merely a pressure below atmosphere. We will return to Pressure Decay Testing frequently during this course.

## *Trace Gas Sensing*

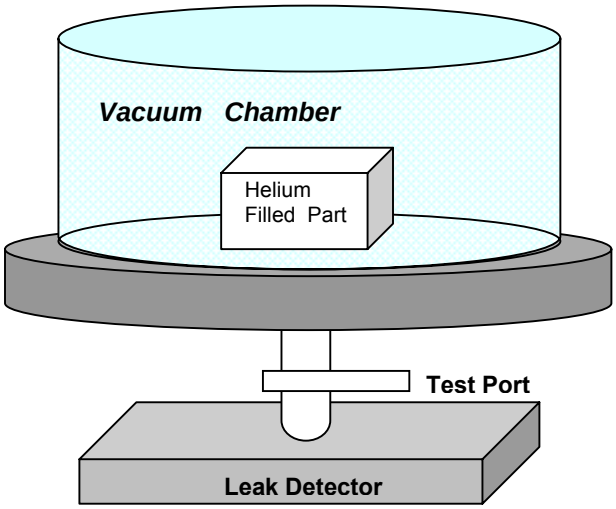



There are several types of trace gas sensing systems on the market today. This diagram illustrates a simplified trace gas leak detector. The closed test part is pressurized with a tracer gas, and the sensor is moved around the part to determine if and where there is a detectable leakage of the tracer gas. A constant flow pump allows measurement of varying concentrations of gas. Of course, using this detector as a leak locator is easy, but using it as a quantitative leak detector requires skill! This instrument is also available for hazardous gases and work areas (intrinsically safe). This method gives quantitative measurements so standards can be precisely described. It is straightforward and reasonably easy to use, and the cost is low. However, because it is dependent on sensing a gas other than air, gas sensing is not useful for sealed packages that cannot be pressurized with a trace gas.




## *Mass Flow Sensing*

The Mass Flow test method uses a small sensor that heats air and then measures the change in temperature with regard to mass flow. Since mass flow is a function of the density of air, care must be taken to check that the calibration is for sea level pressure, otherwise small differences may occur from place to place. The mass flow detector is less sensitive to temperature changes than pressure decay. Mass flow meters vary in their response time depending on the level of leak rate; low rates can be longer especially if maximum values are exceeded.

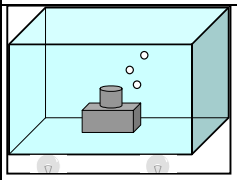
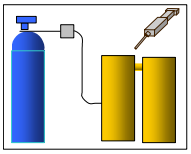
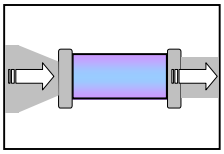
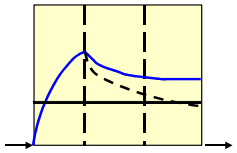
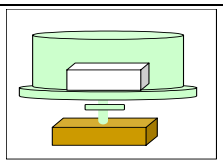



### *Mass Spectrometry*

Mass Spectrometry is the most sensitive of all test methods, being capable of detecting $10^{-11}$ sccs using helium. The helium-pressurized test part is placed in a chamber which is evacuated to a vacuum of $10^{-5}$ millibars, and any helium leakage is drawn into the mass spectrometer tube. The advantages to mass spectrometry include a very high level of sensitivity and a high degree of quantitative accuracy. However, mass spectrometry is extremely expensive, with equipment costs in the range of $25,000 - $100,000. The method is slow, and the cost to run and maintain the equipment is significant.

## *Types of Leak Tests – Advantages and Disadvantages*

Below is a brief summary of several commonly used test methods, including advantages, disadvantages and the relative price of “No Leaks”.

| | | ADVANTAGES | DISADVANTAGES | APPROXIMATE SENSITIVITY | RELATIVE COST |
|---|---|---|---|---|---|
| Bubble (“Dunk”) Testing |  | Simple<br>Inexpensive | Operator dependent/Non-quantitative | $10^{-2}$ to $10^{-3}$ sccs | $100-$1,000 |
| Trace Gas Sensing |  | Low Cost,<br>Best as leak locator | Not usable for sealed packages | $10^{-4}$ to $10^{-5}$ sccs (helium) | $3,000-$10,000 |
| Mass Flow Sensing |  | Fast response<br>Quantitative | Ambient air pressure sensitive | $10^{-2}$ to $10^{-3}$ sccs | $4,000-$10,000 |
| Pressure Decay Testing |  | Quantitative<br>2-4 sec. Tests | Sensitivity is dependent on part size and test time | $10^{-4}$ to $10^{-6}$ sccs | $5,000-$12,000 |
| Mass Spectro-metry |  | Extremely sensitive | Slow, high cost to run and maintain | $10^{-9}$ to $10^{-11}$ sccs | $25,000-$100,000 |

## *Unit 3: Pressure or Vacuum Decay Leak Testing*

### *The Test Cycle*

#### *Charge, Stabilize, Test and Handling Times*

#### *Two Types of Leaks: Gross or Fine*

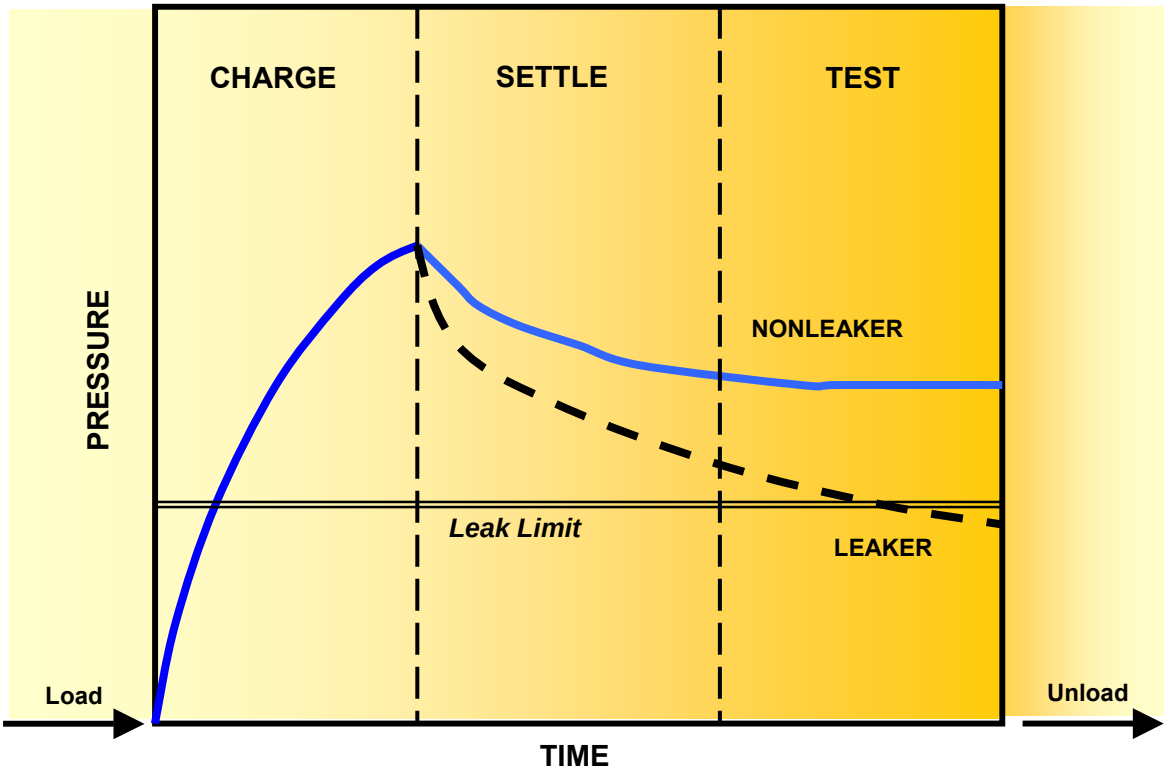
### *Choosing a Leak Rate*

### *Examples of Pressure/Vacuum Decay Leak Testers*

## *Pressure Decay Leak Test Cycles*

What is happening during a leak test? The leak test cycle is actually broken down into three distinct phases, not counting the load and unload phases. The following diagram illustrates the relationship between these phases.

***The Leak Test Cycle***




***Load and Unload*** are the times it takes to engage and disengage your part or package from the pressurizing and pressure decay measuring instrument. Although not technically part of the actual test cycle time, these periods must be taken into account in order to realistically project the time needed to test individual items.

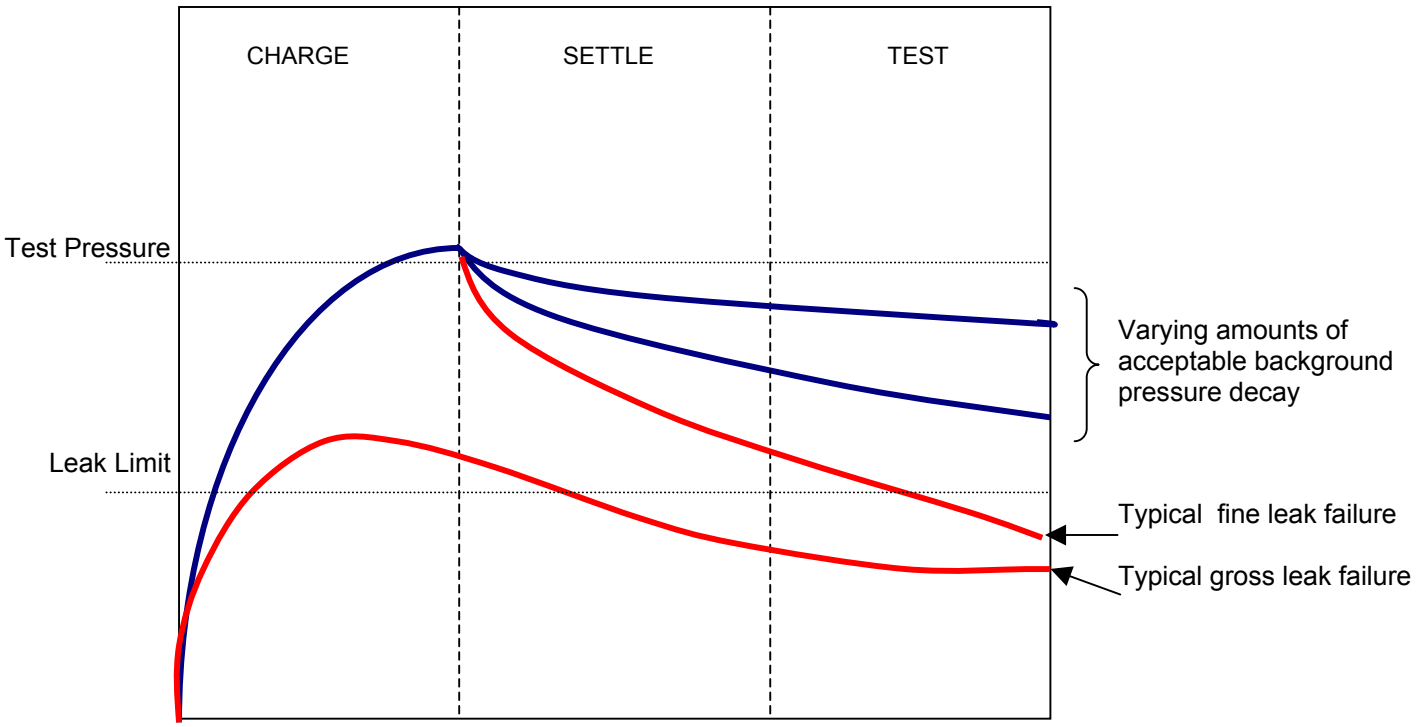
***Charge*** is the period of time in which the part is being pressurized to the predetermined test pressure (or slightly above this pressure, so any stability changes can be taken into account).

***Settle*** is the period allowed for the volume of the pressurized part or package to change and stabilize due to the stresses introduced by pressurization. This is particularly crucial in the case of flexible materials whose volume may change substantially with pressurization. If this is an issue for your product, you will want to review the discussion on restraining plate fixtures in a later unit of this course. The Settle period also allows time for the adiabatic temperature rise (the heat generated through compression of a gas) to stabilize.

***Test*** is the data taking period in which the measurement of the decay of pressure is taken.

### *Two Types of Leaks*

***Decay*** is a term used for the difference in pressure from its initial state of complete pressurization to the pressure at the end of the "test" phase of the leak test cycle previously discussed.




Once pressurized and stabilized, the test instrument will measure the decay of the pressure inside the test part or package over a predetermined period of time. As we have already learned, everything leaks; there will be some amount of apparent background leakage, even if only molecules permeating a rigid container. This pressure decay becomes more significant as the test time increases. This decay may be related to the physical or mechanical properties of the product as well. This tendency is an important consideration when setting the *Leak Limit.*

If the part or package you are testing possesses a *gross leak,* as for example an unsealed joint, that prohibits complete pressurization, the test may not complete the Charge phase of the test cycle, and the test may be terminated at this point.

If the part being tested can be completely pressurized but the decay of the internal pressure is greater than the expected background decay for that part, it may indicate an unacceptable level of leakage. This is a *fine leak*. The selection of an appropriate Leak Limit for the product being tested will differentiate between "normal" background pressure decay and an unacceptable fine leak in the test item.

## *Pressure Decay Testing: Choosing a Leak Rate*

### *Setting the Leak Limit*

The Leak Limit is set based on your need for sensitivity in detecting a pressure decay in your part or package that exceeds what has been determined to be "normal", acceptable leakage. You want to convert your pressure decay (∆P) to leak rate. The guiding equation is:

$$Q\ (sccs) = \Delta P\ (atm) * V\ (cc) / \Delta t\ (sec)$$

…where ***Q***, leak rate, is expressed in standard cubic centimeters per second;
…where ***t*** is the test time in seconds;

*The longer the test time, the smaller the leak rate you will be able to identify. Remember, we agreed that everything leaks; it's only a matter of time! When considering the test cycle for production times, don't forget to include all phases of the test cycle (Load, Charge, Settle, Test and Unload) – see the unit on Pressure Decay Test Cycles for more information.*

…and ***V*** is the internal volume of the pressurized system.

*Pressure decay sensitivity is also a volume function. When selecting your spec, remember to include all the volume in your test system – the instrument, the fixture and your part – in your calculations.*

To maximize the sensitivity of your test, it is also important to consider the stability of your instrument and its environmental conditions such as ambient temperature and temperature changes. However, most test cycles are short in seconds and temperature change may be negligible.

**EXAMPLE**

Let's consider an example. You have performed a pressure decay test on your part and found the pressure decay (∆P) to be 0.004 psi (.1 in$H_2O$). This translates to $2.5x10^{-4}$ atm. Your part, plus the internal pressurized volume of the instrument and the fixture, has a volume of 100cc. Your test time was 10 seconds. Using our formula above, we calculate:

**Leak Rate = $2.5 \times 10^{-4}$ atm * 100cc / 10 sec = $2.5 \times 10^{-3}$ atm-cc/sec**

This is the leak rate for your part. Is it acceptable or a reject? That is a decision you must make as you set your Leak Limit. If this is your Leak Limit, then it is the maximum acceptable leak rate for your part and your Pressure Decay Test should reject any parts with a greater pressure decay at this test time.

## *TM Electronics, Inc. Leak and Flow Testers*

### *The TME SOLUTION© Leak & Flow Tester*


The TME *SOLUTION* is a high resolution leak and flow tester featuring one to four channel concurrent or multiple channel sequential leak and flow testing. Sensitive, repeatable and reliable, the *SOLUTION* can be configured to perform ten different tests on product, including burst, occlusion, vacuum and pressure decay, crack, and differential pressure or vacuum. Touch screen menu-drive operation allows the operator to control the test parameters, examine statistical analysis of results or download data files easily. The *SOLUTION*, in conjunction with custom fixtures, accessories and engineering support, provides a complete turnkey solution to your leak and flow testing problems.

**The TME INDUSTRIAL *SOLUTION*© is available in a NEMA-4 enclosure for harsh environments.** All TME *Solution* models are available with Ethernet capability.




### *The TME WORKER© Leak, Leak/Flow or Leak/Occlusion Tester*

The TME *Worker* is an affordable, every-day, high-resolution (0.0001 psig) test instrument available as a pressure or vacuum tester. The *Worker* has a small footprint, fast response, and is easy to program and use. PLC controls for semi-automatic operation and two-way RS-232 communication capability for downloadable program selection and uploadable data is standard, and Ethernet connectivity is optional.

## *Unit 4: Mass Flow Testing and Flow Testing for Occlusions/Obstructions*

---

***Mass Flow Testing for Leakage***

***Flow Testing for Obstructions:***

***Mass Flow Testing for Occlusions***

***Back Pressure Occlusion Testing***

***Pressure Drop Occlusion Testing***

## *Mass Flow Testing for Leakage*

Mass flow testing uses intrinsic properties of air to directly measure the amount of air escaping a closed system. A pressure regulator establishes the testing pressure, and then the sensor records any movement of air out of the test system. Mass flows are not affected by temperature changes due to pressurization. Mass flow sensors have limited low range sensitivity, generally useable at greater than one sccm (.02 sccs).

We have seen that either pressure decay or mass flow testing can be used for leak testing. What are the criteria for deciding which is appropriate for your particular need? Depending on the size of the leak you are searching for (and the volume of your product), mass flow leak testing can have several advantages, including speed of test. The mass flow test is not dependent on temperature change, which may be a difficulty for the pressure decay test.

## *Flow Testing for Obstructions/Occlusions*

### *Mass flow testing*

Mass flow testing is available for identifying obstructions in an open-ended test part. Unlike mass flow testing for leakage, mass flow testing for obstructions uses a continual flow measure the blockage in an open-ended device, such as a medical catheter or refrigeration tubing. Once the pressure sensor has indicated that the test part has reached the proper pressure, the flow sensor measures the continuous flow of air through the sensor assembly. Determination of whether the part passes or fails the test is made based on the flow rate through the part. If the flow sensor measures too low a flow rate at the desired pressure, the part will fail the test. Any obstructions in the part, therefore, will restrict the flow of air through the device, thereby causing the part to fail the test.

### *Back Pressure Occlusion Testing*

When a tube or device is pressurized with air, a flow will be established depending on the input pressure. If the flow path is obstructed, more pressure will be required to force flow through the product. Back pressure occlusion testing measures the input pressure to the device. A blockage creates a higher pressure at the device. The instrument measures the input pressure, and limits on the pressure measurement are set in the instrument based on experience with the obstructed part. A higher pressure indicates a blockage.

### *Pressure Drop Occlusion Testing*

A device will often need to vent its pressure in a given period of time for proper function. Alternatively, a part may have a manufacturing defect that creates a flap or one-way obstruction that prevents air from venting the part. Pressure drop occlusion testing pressurizes the part, and then measures the change in pressure when the pressure is removed. The pressure must approach a value or zero in a fixed time. If the pressure does not drop a minimum amount, then the limit set in the instrument will not be met, thus indicating a blockage.

## *Unit 5: TME Statistics Packages: For Quality and Process Control*

TM Electronics' leak testers and package testers contain a standard statistical package that provides not only quality documentation but also process control tools such as control charts, histograms and graphic presentation of each individual test.

Control charts are commonly used to aid in manufacturing process control. The objective of control charts is to monitor the process in real time so if something goes wrong, it can be noted and corrected with the minimum of lost product. The concept behind control charts is as follows:

1. A process "in control" will result in pressure decay test values that fall consistently in a predictable range around the average (see Figure 1). In addition, the average test value will not change appreciably over time when the sealing process is "in control".

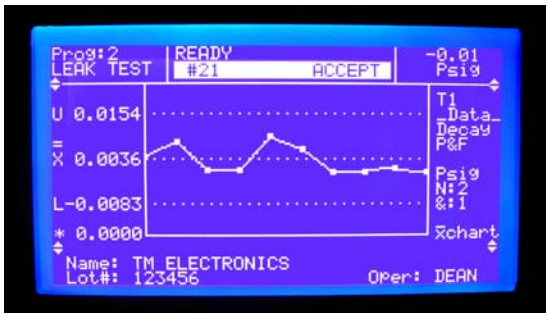



Figure 1 Control charts for leak test results showing a process in control

2. Because processes always vary slightly due to manufacturing and material variations, "good" product test values will go up and down within a range around the mean value. That range can be statistically predicted using the mean test value plus and minus three standard deviations (a measure of the variation inherent in the process). The "acceptable" range is the set of test values that fall between the upper and lower control limits. These control limits are automatically calculated in the TME test instrument from the previous test results in the Datalog.

3. In the TME *Solution*, the data points on the control chart consist of subgroups of test results. These subgroups can be as small as two tests (as in Figure 1), or as many as 20 tests. Subgroups are used to minimize the effect of a testing error or a single bad part.

Control charts for the mean (X-bar) can help the manufacturer in several ways.
If, for example, a temperature problem in your sealing equipment is causing weaker than usual seals resulting in greater pressure decay, the upward trend in test values will be obvious on the control chart even before the product reaches the point of failures. This gives the machine operator an opportunity to correct the temperature problem with little or no loss of product.

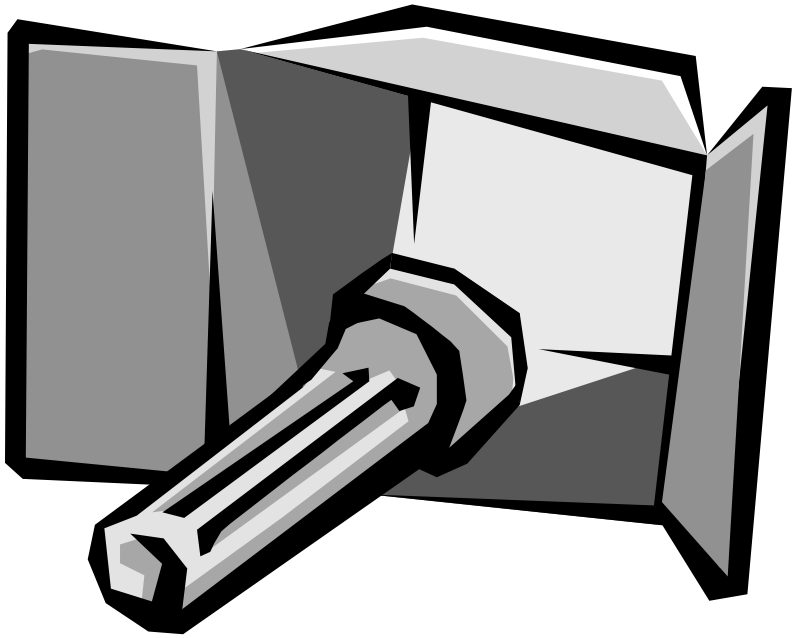
Several data points outside of the control limits (Figure 2) may give the machine operator an indication that instability is developing in the process that needs to be investigated before a large quantity of bad product is produced.

Control charts for range (the difference between the maximum test value and the minimum test value within a subgroup) also have a place in identifying when the process is becoming erratic and inconsistent.

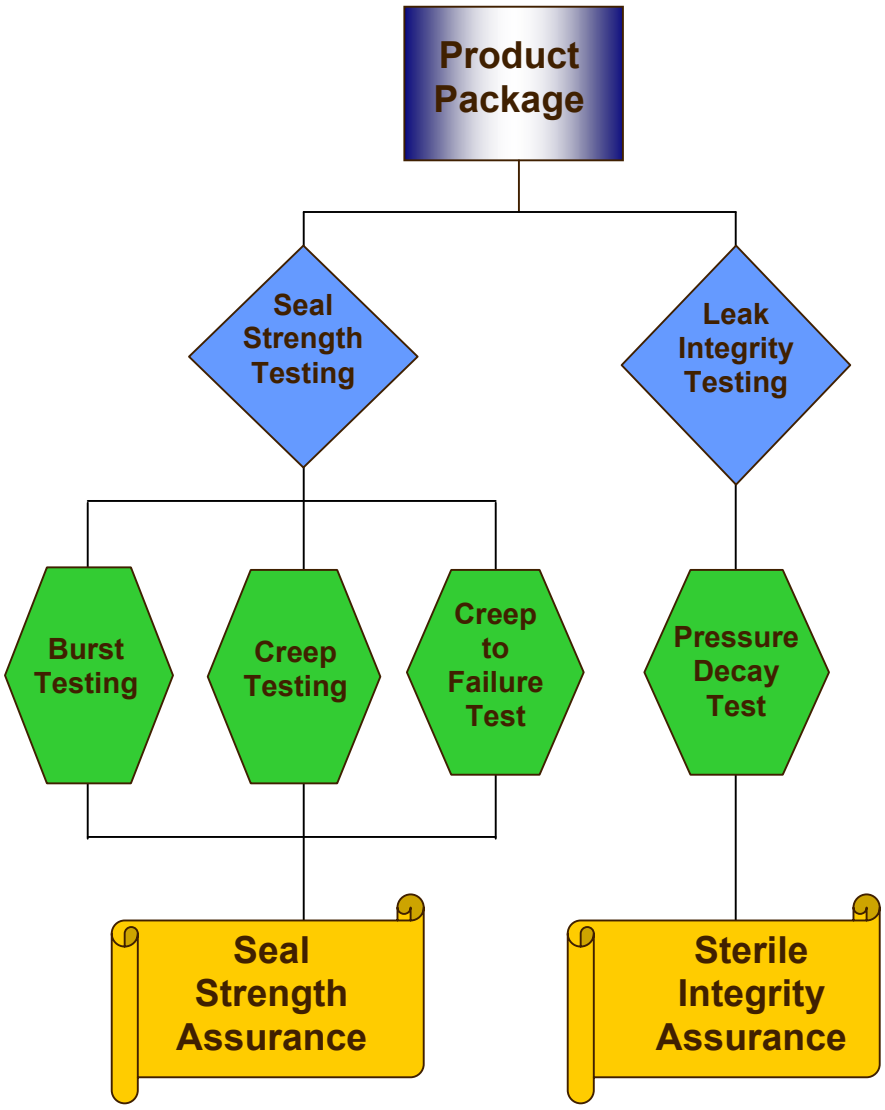

Figure 2 Control charts for process going out of control

## *Term Two:*

## *Package Testing*



# *Second Term Overview: Package Testing*




**Package integrity testing is the next step for the device or product manufacturer.**

There are two roads of testing - seal strength testing and leak integrity testing for non-porous packages.

Seal strength testing is valuable to ensure that package contents don't escape, and that sterile barriers remain intact, under stresses from transport, shelf life and normal usage.

Having completed and documented your device and packaging testing, you can now present your final product to the market.

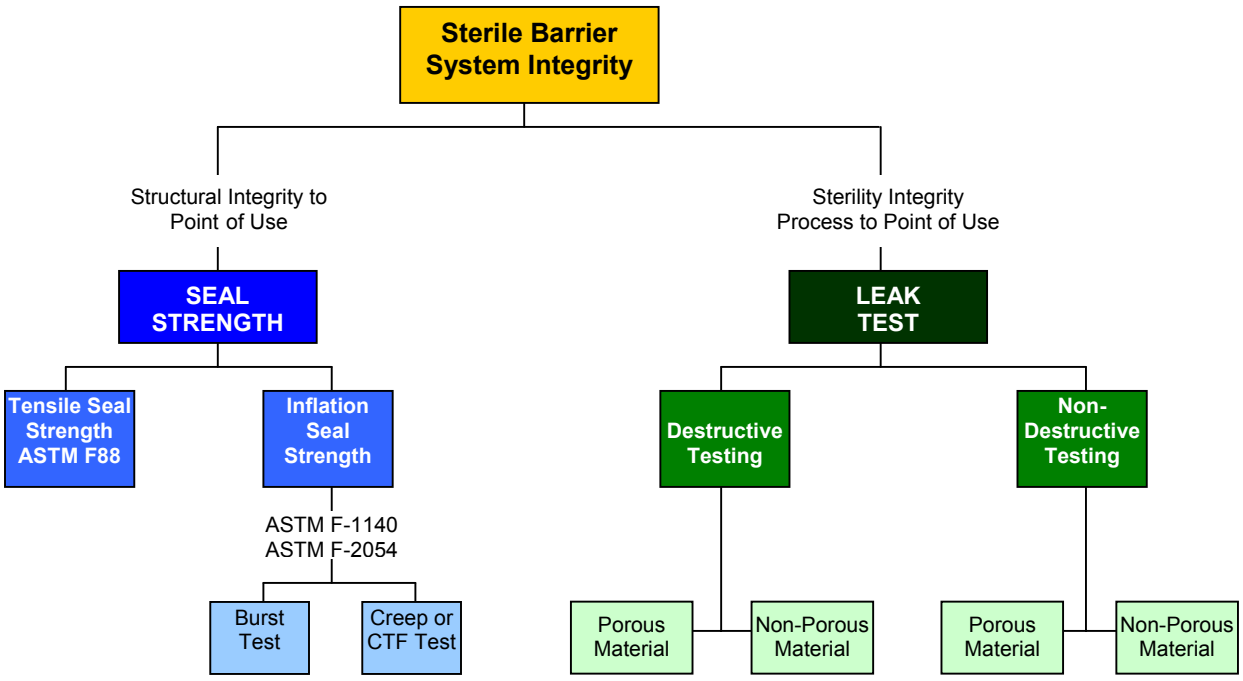
## *Unit 1: Introduction to ANSI/AAMI/ISO 11607 – Essential for Medical Packaging, but Useful and Important Concepts for All*

ISO 11607, "Packaging for Terminally Sterilized Medical Packaging", is an international standard providing a guideline for the design, processing and testing of primary product packages. Because the FDA considers ISO 11607 to be the paradigm for validation protocol for medical device packaging, it is important that manufacturers of these devices rely on guidance from this document as they seek FDA approval of their packaging system validation protocol.

The concepts expressed in ISO 11607 are useful and helpful to non-medical device manufacturers, as well. Whether your product is food for people or pets, electronics, or automotive parts, if you package it, you could find some excellent guidance here.

According to ISO 11607, "the intention of packaging for terminally sterilized medical devices is to maintain the sterility of the product with respect to its intended use, the shelf life, transport, and storage conditions", i.e. to see that the packaging material and process provide a package that will withstand the sterilization and packaging processes and maintain that sterile barrier for the life of the product. This is a twofold objective: first, to ensure the integrity of the sealed package, and second, to assure that no weaknesses in the sealed areas of the package permit leaks to develop with handling stresses and time. To assure that the package performs adequately, you must be sure that the package is able to maintain the integrity of both the seals and the materials under stress. This implies that your package testing system must include both package integrity testing and seal strength testing, two complementary but very different procedures. Package integrity may be thought of as a "leak test" of the package – is there a failure in the materials or process that allows contamination to enter? Seal strength testing, on the other hand, measures an attribute of the seal, which is designed to ensure that the seal presents a microbial barrier to at least the same extent as the rest of the packaging. Both testing streams are important in your final package analysis.

These two paths can be illustrated as follows:



## *Consensus Standards? ASTM Test Methods?*

## A note to medical device manufacturers and packagers:

Before deciding on specific package test methods, it is important to look ahead to the process you, as medical device packaging professionals, will have to deal with: validation of your chosen package test method. What is a validatable test method? A validatable test method is one for which the following characteristics have been defined by ASTM or FDA:

- ***Repeatability***: *what is the variation in results using the same operator, same equipment, in the same location?*

- ***Reproduceability:*** *what is the variation in results with different operators using different equipment in varying locations?*

- ***Sensitivity:*** *what is the smallest value of the tested variable that can be accurately identified by the test method?*

Although you are not required to use a test method for which the above characteristics have already been defined and recognized by FDA, it is greatly to your advantage to do so. If you design your own test method, YOU will be responsible for all the effort that has been done by others, such as ASTM, for validatable methods!

ASTM is a consensus body made up of OEM users, suppliers, instrument and other manufacturers. The ASTM process provides for the development of test methods using a standard procedure, and confirmation of methods using Interlaboratory Studies (ILS). These procedures provide the repeatability, reproduceability and sensitivity data necessary to validate your test method with your product. *FDA Recognized Consensus Standards* are by definition test methods developed by consensus bodies such as ASTM.

For more information about FDA consensus standards, check the following government websites:

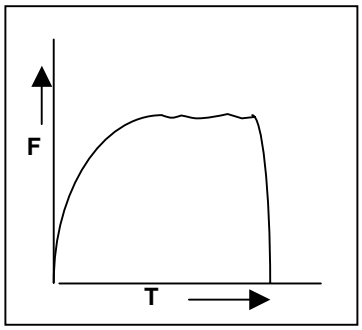
**www.fda.gov/cdrh/osel/guidance/321.html** *for more information;*
**www.fda.gov/cdrh/osel/guidance/109.html** *for FAQ's.*

## A note to the rest of you:

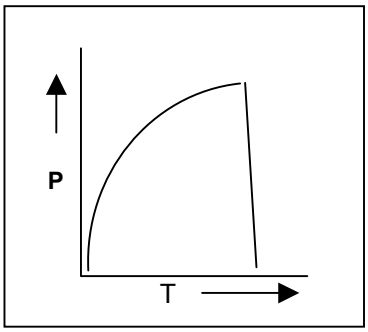
You may or may not be responsible to the government for validation of your test methods for leak or package testing. However, you are of course responsible for providing the best possible protection for your product, your customers, and yourself by selecting the most appropriate test method for your needs. ASTM test methods, when appropriate to your product or package, provide information on repeatability, reproduceability and sensitivity that will enhance your confidence in the way you are testing your product.

## *Unit 2: Seal Strength Testing*

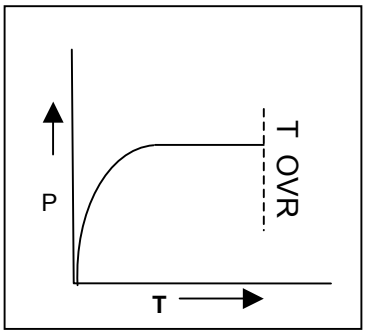
### *Types of Seal Strength Testing*:




Tensile Test



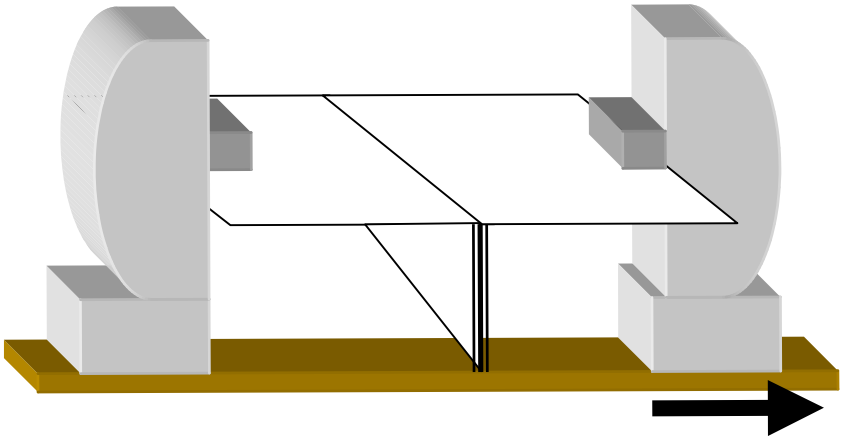

Whole Package Burst Test



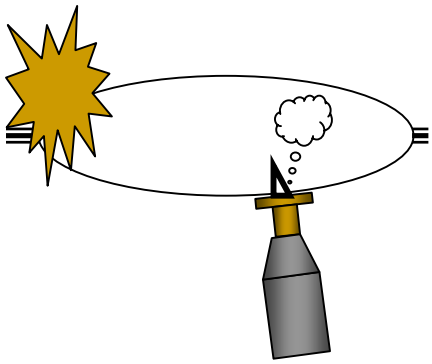

Whole Package Creep Test

### *Tensile Testing*

Tensile seal strength testing measures the ability of a package seal to resist separation – the simple peel strength of the seal. Using a defined width sample of a package perimeter seal, a moving jaw pulls the sample apart at a constant speed while measuring the resistance force during the seal separation. The tensile test is particularly suited to peel-open packages. A significant advantage to this test is its sensitivity, and a disadvantage is that in the majority of cases a perimeter seal is sampled only at several locations and a total package seal strength measure is not obtained. Another disadvantage is that the effect of hoop and lateral stresses from inflation or non-perpendicular peel stress cannot be measured.



## *Inflation Seal Strength Testing*

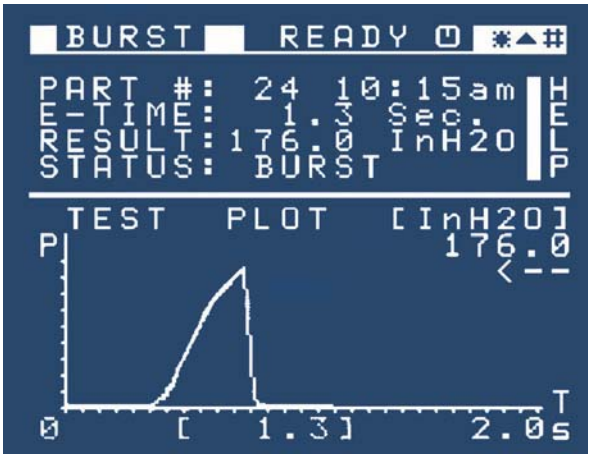


Inflation seal strength testing includes burst, creep and creep-to-failure testing. This test requires pressurizing the entire package and measuring the peak rupture pressure (burst test) or the time to failure at a constantly held pressure (creep and creep-to-failure test). This test provides three 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. This provides a more realistic representation of stresses to which your package will be subject than that provided by the tensile test.

Another advantage to this type of testing is that it provides a whole-package minimum seal strength and also indicates the weakest seal area, and is equally applicable to peelable and non-peelable seals. Inflation tests are applicable to most package forms such as pouches, header bags, lidded trays, flexible or rigid blisters and laminated or rolled tubes.

## *Burst Testing*

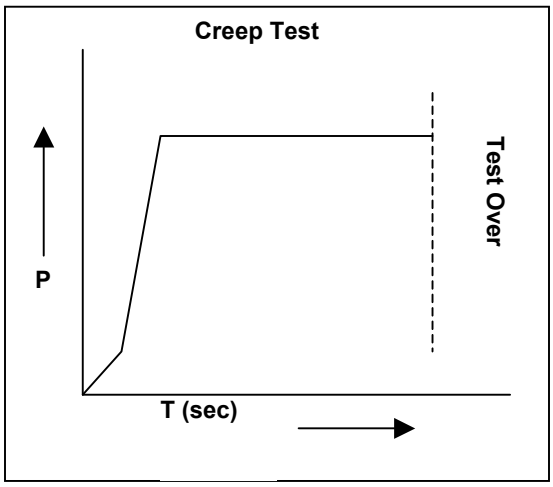
Burst testing determines the overall minimum seal strength of the package seals by inflating the package at a uniform rate until the seal separates at the point of greatest weakness. The graph at left represents the output from the TM Electronics BT-1000 Automated Seal Strength Tester showing a characteristic burst curve. The Burst Test is a peak inflation pressure test; you can see how the pressure increases to a maximum pressure at which the pressure drops to zero. This drop represents the rupture of the seal. The pressure at which the package bursts (176.0 $InH_2O$ on our burst graph) is a variable statistic that can be utilized to document process development and process control through the use of tools such as upper and lower control limits.




Typical Burst Test Graph

Control of inflation rate is important in a burst test to ensure consistent conditions for the test method. The porosity (or lack thereof) of the package material determines the inflation rate for the burst test. Because air escapes through the walls of a porous package during inflation, the flow rate must be increased to compensate for the lost air through the walls and create the back pressure in the porous package.

## *Creep Testing*




The Creep Test is a second general type of whole package inflation seal strength test. In the Creep Test, a whole package is inflated to a constant pressure, which is then held for a specified time, resulting in a pass/fail result. This provides a test for slow shear of the adhesive bond similar to a dead weight hanging on the seal. A suggested starting pressure for peelable seals is to begin evaluating your seal with a creep pressure that is about 80% of the burst value. The inflation rate is not critical, as long as the initial fill is not so fast as to shock the seal or so slow as to result in an overly long test time.

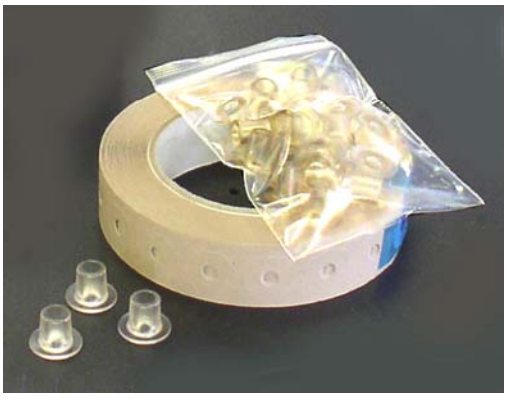
Shortcomings of this test are the need for the operator to visually examine the seal at the end of the test to determine the degree of seal peel, and the lack of a variable statistic upon which to perform process control analysis.

## *Creep to Failure Testing*

This is a variation on the Creep Test that addresses its weaknesses. In the Creep-to-Failure test, the test pressure on the inflated package is held until the seal actually fails, yielding an end point value (a variable statistic), time to failure, and pinpointing the area of greatest weakness in the seal. Time to failure can then be used in SPC or SQC methods.

## *Questions?*

### *How can I get the pressure into the package?*



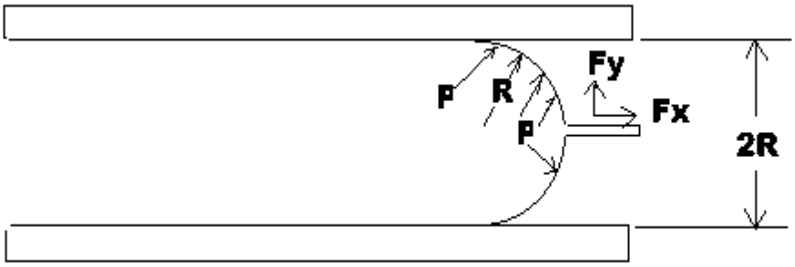
To pressurize a closed package, a leak tight measuring path must be available between the package interior volume and the pressure source. Here we can see a simple, effective method of accomplishing this. This is the TM Electronics' patented Package-Port© system in which a reusable plastic entry port is secured and then accommodates the pressurizing probe. The probe tip pierces the package, enabling pressurization, and the Package-Port reinforces the package material to eliminate any possible leakage of gas around the penetration point. Inexpensive, simple to use and reliable, this system makes in-process package inflation testing highly efficient and repeatable.

## *Unit 3: Restraining Plate Package Testing*

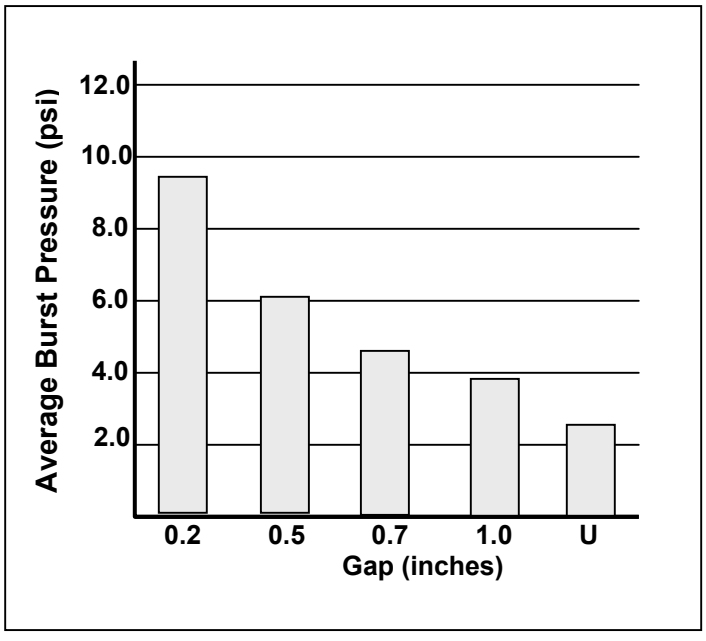
Seal strength values are related to the package size, geometry, and materials. For example, pouches with a long side seal will generally fail on the long seal unless a heater failure has occurred on the shorter seal or chevron. Unsupported tray lid seals may fail at points only relative to their geometry. Very flexible package materials may deform with pressurization to an extent that makes seal testing difficult. To address these problems, it may be advisable to use restraining plates for your inflation testing.

The geometry of the package under test affects the distribution of internal pressure forces on the package surface and seals; for example, a pouch-form package unrestrained in any axis exhibits circumferential hoop stress when internal pressure is applied. When the package is restrained, the load application is distributed directly on the seal area and, because material stretching and deformation is minimized, the test forces are more uniformly applied. The diagram below illustrates the effect of restraining plates on the pouch under test:

where R is the radius, P is the pressure exerted on the unrestrained edges of the pouch, and Fy and Fx are the force vectors on the seal.




Restraining Plate Test Forces on Pouch




In addition, package restraint has a direct relationship to burst pressures: the wider the gap between plates, the lower the average burst pressure (see graph).

The most important factor when interpreting test results is that all conditions in the package test method are *consistent.* Establish a set of test conditions for each package and reproduce those conditions consistently.

Use of package restraints must be approached with caution; because of pressures exerted on the plates, extreme care must be taken that fixtures are designed to withstand the forces applied by the inflated package.


## *Summary – Seal Strength Testing*

**Inflation seal strength** test results can be an excellent tool for process control. Burst test results, creep-to-failure and tensile data are all amenable to use in control charts and provide quantitative data required by ISO-11607 for package testing validation. A number of ASTM test methods, which are accepted FDA Consensus Standards, are available to aid in the design of these tests (see below).

| **ASTM Seal Strength Test Methods** | **Reference #** |
|---|---|
| **Standard Test Methods for Internal Pressurization Failure Resistance of Unrestrained Packages** | F-1140-07 |
| **Standard Test Method for Burst Testing of Flexible Package Seals Using Internal Air Pressurization Within Restraining Plates** | F-2054-07 |
| **Standard Test Method for Seal Strength of Flexible Barrier Materials (Tensile Test)** | F-88-07 |

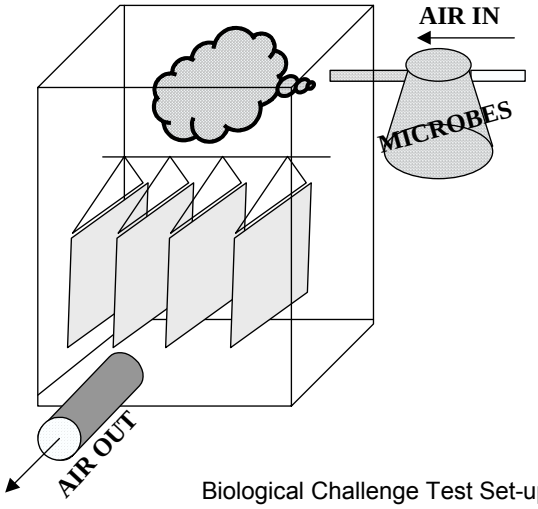
But as we discussed earlier, seal strength testing is only half of the story. The other half is **Physical Package Integrity Testing**.

## *Unit 4: Package Integrity Testing*

We have thoroughly examined the need and methods of seal strength testing. *Package integrity testing* is purely a measure of the package's sterile barrier – a "leak test" of the whole package. In addition to seal bonding failures or disrupted seals, leakage can be the result of large holes, pinholes or cracks in package materials. Either source of leakage represents the potential for product contamination – elements of the ambient atmosphere outside of the package entering the package – or for the materials inside the package to escape.

**Biological Challenge Tests vs. Physical Test Methods**

Biological challenge tests, in which the package is isolated in a chamber, surrounded by microbe-laden atmosphere, and then examined after the microbes have been removed, are indicated by ISO-11607 for finished medical device packaging. Recent studies indicate that these high bio-burden aerosol tests may not be the most reliable indicators of leakage. Physical test methods, which are more reliable and more repeatable, present the best opportunity for determining the integrity of the package.



Biological Challenge Test Set-up.

The ISO-11607 List of methods for physical integrity tests includes these methods:

*Visual Inspection Method*
*Internal Pressure Method*
*Vacuum Leak Method*
*Dye Penetration Method*

These methods have been reviewed and are recommended for use. A drawback of these methods is that they are not quantifiable, and that they require operator interpretation.

The FDA has accepted certain test methods produced by industry consensus organizations as being valid for medical device packaging. This means they have been evaluated for repeatability, reproduceability and sensitivity, and make the validation process much easier for the medical packaging professional. These consensus standards are also an excellent place to start for those outside the medical packaging community as they begin the process of designing or selecting a package test method. The table below gives an overview of currently used package test methods, their ASTM test method designation if applicable, appropriate usage circumstances, advantages and disadvantages.

**Note: It is very important that you think about your package, your process and your end product's needs when evaluating test methods. Among other things, you should consider:**

- **Are your barrier materials *porous* or *non-porous?***
- **Will you need to test the whole package, or just the seals?**
- **Is a *destructive* test or a *non-destructive* test more suitable to your needs?**
- **Are you able to consider an expensive test, or are you operating on a budget?**

## *The Alternative to Microbial Challenge: Physical Tests with ASTM Test Methods*

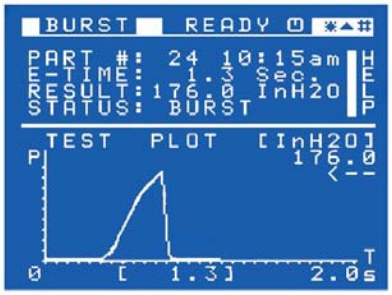
| Test Method | Package Form | Barrier | Destructive Or Non? | Cost | Sensitivity |
|---|---|---|---|---|---|
| **Dye Penetration (Blue Dye Test) ASTM F-1929** | Pouch, tray | Porous or Non-porous | Destructive | $ | 50 um (.002") channel leak |
| *Advantages: Inexpensive, commonly used test* | | *Disadvantages: Messy, operator dependent, requires clear material on at least one side, difficult to use with papers and some porous materials that can absorb dye; qualitative* | | | |
| **Visual Inspection ASTM F-1886** | Pouch, tray | Porous or Non-porous | Destructive | $ | 75 um (.003") channels |
| *Advantages: Inexpensive, convenient* | | *Disadvantages: Dependent on operator, materials, magnification etc.; can't rule out pinholes; qualitative* | | | |
| **CO2 Tracer Gas ASTM F-2228** | Trays with porous lids | Porous | Non Destructive | $$$ | 100 um channel 50 um hole |
| *Advantages: Not operator dependent; will find leaks in rigid tray material as well as channels in the seal* | | *Disadvantages: Not considered a whole package test; does not challenge the porous component of the package, limited use* | | | |
| **Bubble Emission ASTM D-3078** | Flexible material | Non-Porous | Destructive | $$ | 100 um hole or 1 x 10-5 cc/sec |
| *Advantages: Useful for gross leak detection; useable with a variety of package forms* | | *Disadvantages: May not detect small leaks; dependent on product contained, materials etc; Operator dependent, messy, qualitative* | | | |
| **CO2 Tracer Gas ASTM F-2227** | UNLIDDED Rigid Trays | Non-Porous | Non Destructive | $$$ | 50 um (.002") pinholes |
| *Advantages: Not operator dependent* | | *Disadvantages: Not for whole package, applicable to empty trays only* | | | |
| **Pressure Decay with/without** Restraining Plates ASTM F-2095 | Flexible pouches, Foil sealed trays | NonPorous | Destructive | $$ | 25 um pinholes or 1 x 10-4 sccs |
| *Advantages: Quantitative, not operator dependent, fast, easy to use, wide range of applicability* | | *Disadvantages: Sensitivity dependent on package volume; doesn't show location of leaks; not amenable to porous barriers or liquid contents* | | | |
| **Bubble Test (Internal** Pressurization) ASTM F-2096 | Flexible material | Porous or Non-porous | Destructive | $ | 250 um hole |
| *Advantages: Useful for gross leak detection; useable with a variety of package forms; inexpensive* | | *Disadvantages: Messy, operator dependent, use with porous in certain circumstances only, long test time* | | | |
| **Vacuum Differential ASTM F-2338** | Trays (lid/no lid) and cups | Porous or Non-porous | Non Destructive | $$$ | Varies with application |
| *Advantages: Not operator dependent, amenable to a variety of package forms and materials* | | *Disadvantages: Requires sealing of porous surfaces* | | | |
| **Helium Tracer Gas** ASTM F-2391 | Flexible or Rigid Packages | Non-Porous | Non Destructive | $$$$ | 10-10 sccs |
| *Advantages: Quantitative, can detect moderate ("Sniffer Mode") to very fine ("Vacuum Mode") leaks* | | *Disadvantages: Expensive, nigh maintenance* | | | |

### *Some Other Alternatives: Physical Integrity Tests in Use*

| Test Method | Package Form | Barrier | Destructive Or Non? | Cost | Sensitivity |
|---|---|---|---|---|---|
| **Pressure/Vacuum Decay Chamber Test** | Sealed, flexible material | Non-Porous | Non-Destructive | $$$ | 5-10 um hole |
| *Advantages: Quantitative, fast, not operator dependent, can be semi-automated or manual* | | *Disadvantages: Not a consensus standard; requires custom fixture for greatest sensitivity* | | | |
| **Ultrasonic Leak Detection** | Pouches, flexible packages | Porous or Non-porous | Non Destructive | $$$$ | 25 um |
| *Advantages: No sample preparation; detects non-leaking seal defects; fast* | | *Disadvantages: Not a consensus standard* | | | |
| **Force Decay** | Pouches | Non-Porous | Non Destructive | $$$ | 25-50 um |
| *Advantages: Fast, quantitative, not operator dependent* | | *Disadvantages: Not a consensus standard; requires custom fixtures for greatest sensitivity* | | | |

## *TM Electronics Package Testers*


**TME'S BT-1000 Package Tester** is versatile, automated and able to perform both leak-integrity tests and a battery of seal-strength tests on food, medical device and pharmaceutical packages. Foil, laminate and barrier packaging materials are well suited for the burst, creep and creep-to-failure seal strength tests, as are porous pouches, trays and blisters with materials such as Tyvek® and paper barriers. The BT-1000 features eight test modes for conducting both types of testing on the same nonporous package without having to change the setup, test item or instrument settings. Graphical results of individual tests and SQC analysis of readings in the data log optimize process control.




The BT-1000 graphically shows the characteristic burst curve for each test.


Accumulating test data are shown in histogram form to give a visual overview of your process.


Ongoing test data are shown on a statistical control chart for tighter process control.

SQC is also a trademark of the TME **Solution-C Non-Destructive Package Tester,** which provides fast, nondestructive testing of flexible packages and real time statistical analysis and quality control charts. The Closed Chamber Test Method allows a pressure decay test instrument to look for changes in pressure due to air leaking into or out of a surrogate test chamber volume using either pressurization or vacuum techniques on the package under test.

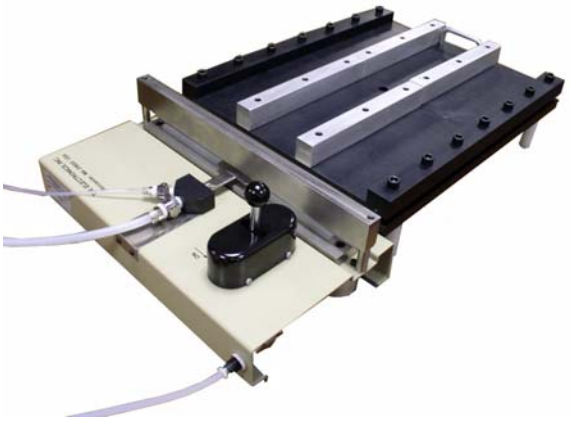

Holes as small as 5 to 10 microns can be detected in pre-formed lidded trays, blister packs or pouches of common package materials including films, foils and laminates. In-process leak testing will detect leaks from pinholes, cracks, seal and channel leaks without disrupting your production process. Proprietary test techniques provide identification of large ("gross") leaks as well as small leaks.

## *TM Electronics Package Testing Fixtures*

This **Closed Package Test Fixture,** in conjunction with an entry system such as the TM*Electronics* **Package-Port© system,** allows you to test flexible package Seal Strength and Leak Integrity on completely sealed packages. This fixture can accommodate packages up to 12" wide (with probe down) and up to 24" wide (with probe up). A variety of probe sizes is available to further customize your package integrity test system.

The **Open Package Test Fixture** provides a pneumatically driven clamp that will seal the open end of a flexible package during the inflation testing for package seal strength in accordance with ASTM F-1140. Both 12 and 24-inch clamp bar models are available, for most medical, food and industrial packages.

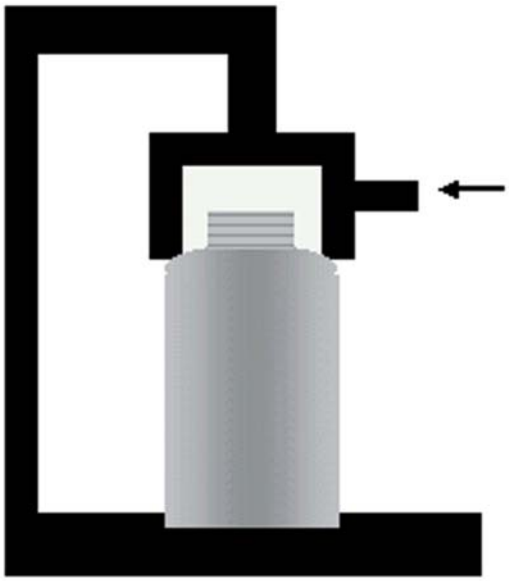
**Restraining Plate Fixtures** (below) for leak testing pouches have semi-porous surfaces to stabilize expansion during pressurization without blocking any holes in the surface material. Restraining plate fixtures for seal strength tests provide consistent stress loading on all seals. The restraining plates can be combined with the open package test fixture to enable restrained testing on sealed packages. Use of restraining plates is supported by ASTM F-2095.

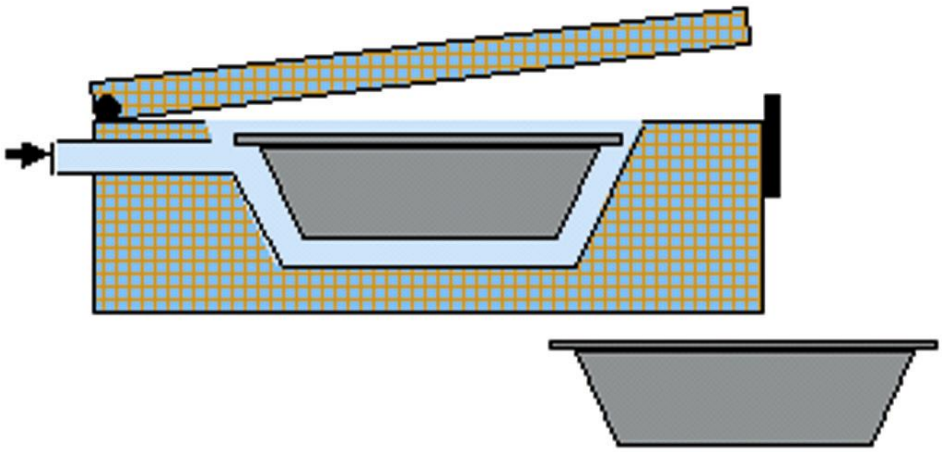


Modifications are available from TM Electronics to customize your package testing system to accommodate your particular product. Contact the Engineering Department at TME for more information.

## *Term Three:*

## *Non-Destructive Pressure/Vacuum Decay Chamber Testing For Sealed Products or Packages*





## *Unit 1: Surrogate Chamber Testing using Pressure or Vacuum Decay*

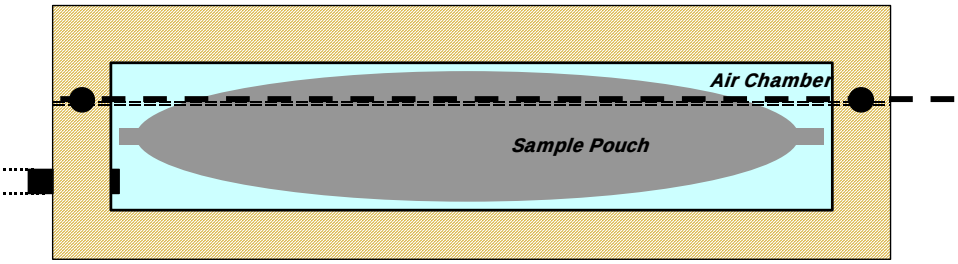
In the course of looking at leak and package testing methods, we have looked briefly at several non-destructive test methods, including:

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

We are going to focus more deeply on the latter. 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.

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 pre-formed lidded trays, blister packs, or pouches.

**Introduction to Surrogate Chamber Testing Using Pressure/Vacuum Decay**

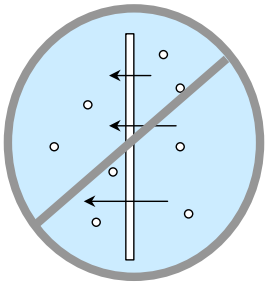



Traditional pressure decay testing supposes a test item or package that can be pressurized. If your product or package is closed or sealed so it cannot be pressurized from an external source, an alternative and non-destructive method of pressure decay leak testing involves creating a closed space around 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.

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, though, that 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 $10^{-4}$ sccs is an achievable sensitivity. The method is quantitative; your test results are amenable to SQC analysis for process control.

## *Unit 2: Understanding your Test Item*

### *1. What materials comprise your test item or package?*

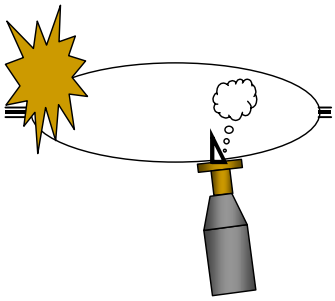


Materials (package walls and seals, closed product surfaces etc.) must be non-porous to air movement. Examples of packaging materials suitable for chamber testing are:

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

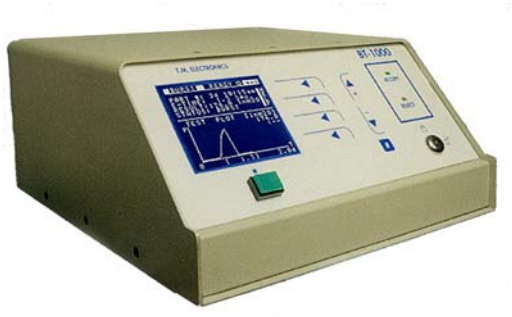
Paper and Tyvek® are not suitable for this type of testing, as they are porous to air movement. Examples of closed products suitable for chamber testing include closed ended extrusions, vials, bottles, and welded housings.

### *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. 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. 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.

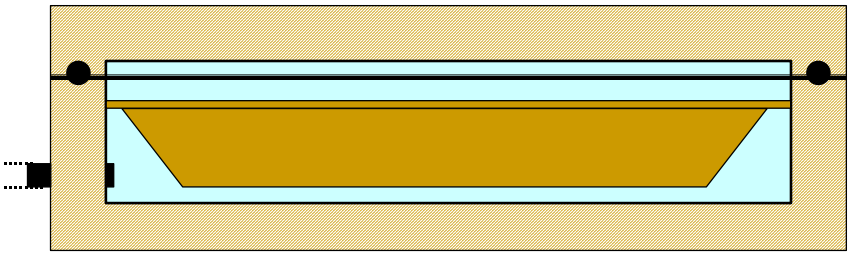
### *Establishing your Seal Strength*



TME'S BT-1000 Package performs both seal strength and leak integrity testing. The BT-1000 is the ideal instrument to help you understand your seal strength characteristics.

The BT-1000 features eight test modes for conducting both types of testing on the same nonporous package without having to change the setup, test item or instrument settings.

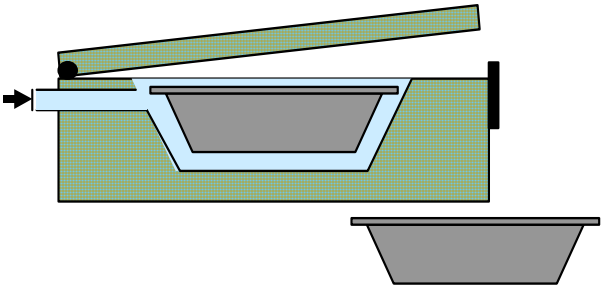
## *Unit 3: Designing your surrogate chamber*



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.

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***

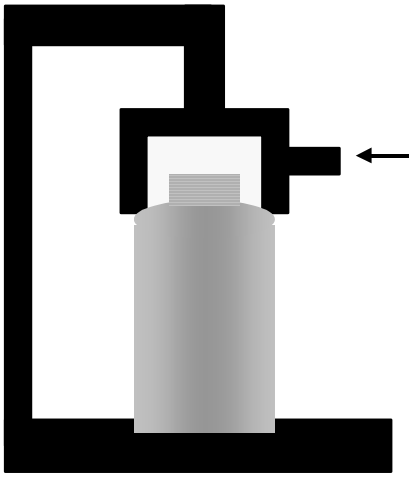


***Typical chamber fixture to accommodate pre-formed, filled and lidded trays***

*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.***

## A Caveat: Adequate Head Space 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.

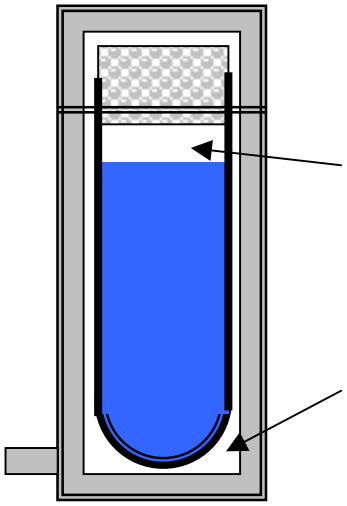


**Diagram of Test Chamber for Liquid-Filled Vial**

**Head space (inside vial)**

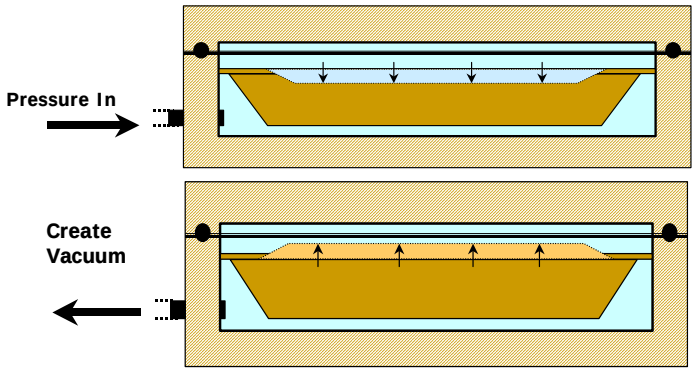
**Interstitial space (outside vial, inside sealed chamber)**

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.

TM Electronics engineers evaluate each potential application of chamber testing technology to assure that this condition is met.

### *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.

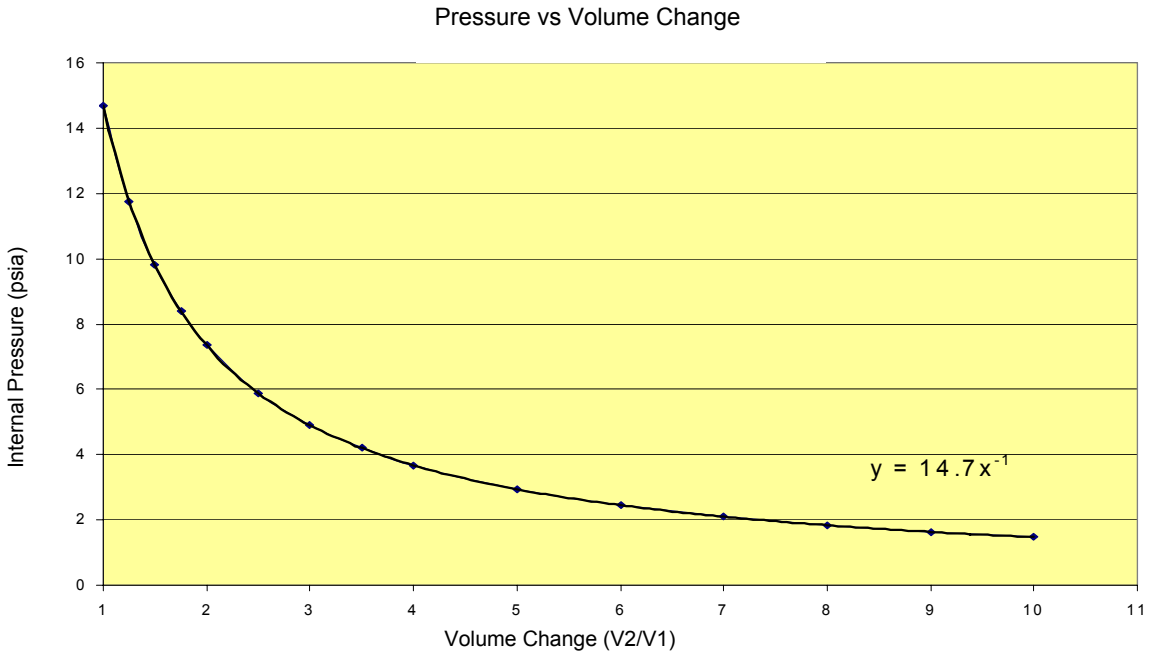
### *Why is Pressure Decay the preferred method?*

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. 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:

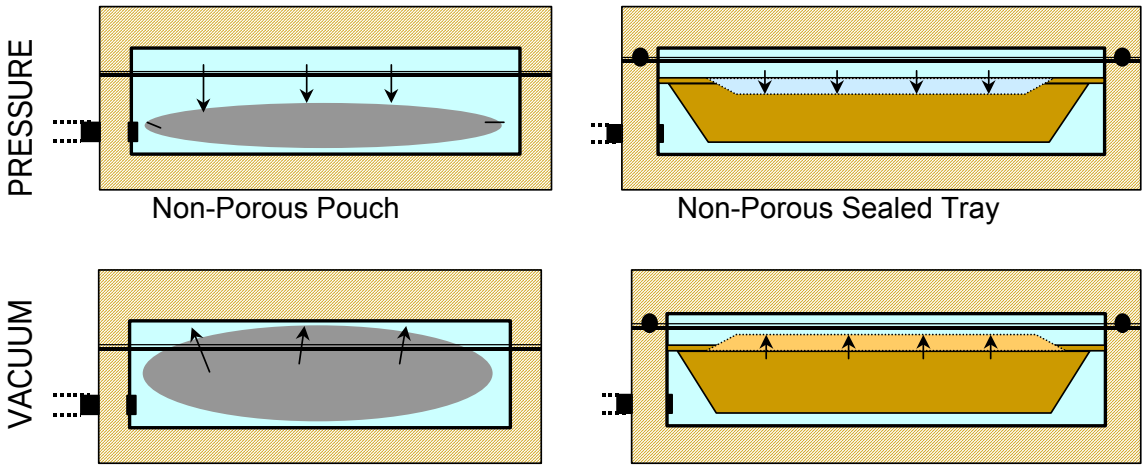
$$P_1 V_1 = P_2 V_2$$

which we can see in the graph.




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.

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 Page 3) and substitute the interstitial volume $V_C$ for the system volume V, you can see the chamber leak effect:

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

where Q is the leak rate, Δ P is the change in pressure in the chamber after stabilization, and Δ t is 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.



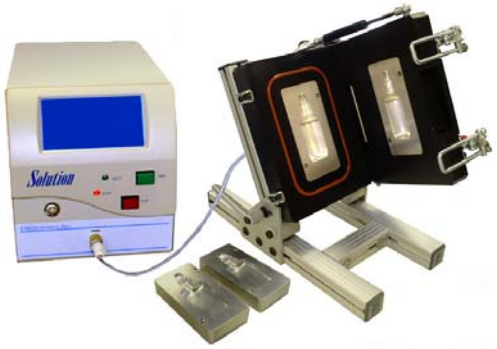
TME *Solution-C* Two Port Chamber Tester with chamber fixture custom designed to non-destructively test automobile accessories

## *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, nutriceutical 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 Test 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. Contact TM Electronics to discuss your application.

## *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, $kg/cm^2$ 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 product meeting predetermined specifications.

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

# BIBLIOGRAPHY AND REFERENCES


AAMI TIR No. 22:2007. "Guidance for ANSI/AAMI/ISO 11607-2007, Packaging for terminally sterilized medical devices." ©2007, Association for the Advancement of Medical Instrumentation, Arlington, VA 22201.

ANSI/AAMI/ISO 11607 Part 1 & 2, "Packaging for terminally sterilized medical devices." 2007, International Organization for Standardization.

"ASTM F1140, Standard Test Method for Failure Resistance of Unrestrained and Nonrigid Packages for Medical Applications," *ASTM Standards, Section Fifteen,* vol. 15.10, West Conshohocken, PA, American Society for Testing and Materials, 2007.

"ASTM F2054, Standard Test Method for Burst Testing of Flexible Package Seals Using Internal Air Pressurization Within Restraining Plates," *ibid.*

"ASTM F88, Standard Test Method for Seal Strength Testing of Flexible Barrier Materials," *ibid.*

"ASTM F2095, Leak Tests for Non-Porous Packages", *ibid.*

*Nondestructive Testing Handbook,* 2nd Ed., Vol. One, "Leak Testing". R.C. McMaster, Editor, ASNT, Columbus, OH 1982.

FDA, *Guideline on General Principles of Process Validation,* USFDA_CDER, May 1982; see http://www.fda.gov/dcer/guidance/pv.htm

FDA, "Recognition and Use of Consensus Standards; Final Guidance for Industry and FDA Staff", issued June 20, 2001; see www.fda.gov/cdrh/ost/321.html.

Minimum Channel Leak and Pinhole Size for Bacterial Entry into Polymeric Trays...; Pascall &Teo, IIT; 2000 IFT, Session 21-1, 2000

*Nondestructive Testing Handbook,* 2nd Ed., Vol. One, "Leak Testing". R.C. McMaster, Editor, ASNT, Columbus, OH 1982.

Plastic Package Integrity Testing; Blakistone & Harper Editors; IOPP & FPI, 1995

Pharmaceutical Container/ Closure Integrity I- IV; Kirsch, U of Iowa; PDA Journal, Vol 51, No. 5, 1997 & 53, No. 4, 1999

***Publications available at www.tmelectronics.com:***

Franks, Stephen H. and Donald S. Barcan. "Examining the Relationship of Tensile and Inflation Seal Strength Tests in Medical Pouches". ©1999, Donbar Industries, Inc. & TM Electronics, Inc.

Franks, Stephen. "Calculating Factors of Safety for Package Burst and Creep Test Fixtures". *Medical Device & Diagnostic Industry,* June 1998.

Franks, Stephen. "Leak and Flow Testing 101". ©2000, TM Electronics, Inc.

Franks, Stephen. "Strength and Integrity – the Basics of Medical Package Testing". ©2002, TM Electronics, Inc. (as published in *Pharmaceutical and Medical Packaging News)*

*This mini-course has been presented by*

# TM*Electronics, Inc.*

## *Specialists in Leak, Flow and Package Testers*


45 Main Street
Boylston, MA 01505

Phone: (508) 869-6400
Fax: (508) 869-9955

E-mail: **sales@tmelectronics.com**


Visit us any time at our website:

**www.tmelectronics.com**

***We can tell you a lot…***
***about your product!***