Designing Package Closures that are Easy to Test with High Confidence:

A few obscure principles that affect the leak tester, with examples of closures that are easy to test, and a few that are not.

Paul B. "Brad" Shaw.

Leak Testing Specialists, Inc., Orlando, FL, USA

ABSTRACT

There are specific design details that can make a radioactive material package closure easy to leak test quickly, economically, and confidently. However, there are also details that can make it difficult to test. These difficulties cause the operation to be more time consuming to complete, with results that have a lower test confidence. This paper will present a number of principles and design features that make for robust package closures that are economical to leak test with a high test reliability. The paper will include specific examples that may be of benefit to the designer, the package owner, and the user.

INTRODUCTION

The closures of some packages for transportation of radioactive materials can be leak tested to the required test sensitivities with relative ease. The leak test procedures for these packages can be qualified with minimal effort. In production, the leak tests of these packages can be performed under a wide range of environmental conditions, with comparatively consistent schedule duration.

The closures of other packages for transportation of radioactive materials are difficult to leak test to the required test procedure sensitivity. The effort required to qualify the leak test procedures for these package closures can be time consuming and expensive. The leak tests of these packages may require that the environmental conditions in the area of the test be tightly controlled, and the process time required for leak testing these closures may vary widely, leading to substantial expense due to the uncertainty of the schedule.

This paper will present six (6) design considerations that have contributed to the production of RAM package closure designs that can be readily leak tested to the required test sensitivity, with relatively standard test procedures, with consistent production time durations, under a comparatively wide range of environmental conditions.

PROVISION OF ADEQUATE CONDUCTANCE IN THE CLOSURE TEST PATH

The conductance of the fluid path in the closure and the test port access to the closure are a significant concern for achieving package closures that can be easily tested. When the package closure is designed with an inadequate conductance, leak testing of the closure to the required test sensitivity may be difficult, unreliable, or impossible.

Tracer gas leakage rate tests of package closures that include an elastomer element must typically be completed quickly enough to allow leakage rate measurement before the permeation of the elastomer element interferes with the leakage rate test. Package closures with inadequate conductance for leak testing will require excessively long test dwell times. The long test dwell time may make it difficult or impossible to complete high sensitivity leakage rate tests without encountering tracer gas permeation of the elastomer closure element.

Of particular concern are package closures that must be tested by leakage rate test methods that are dependent on the molecular flow mode of fluid flow.

The molecular mode of fluid flow is not taught in traditional Engineering Fluids courses, but it is particularly important in the design of package closures that must be tested by means that involve evacuation (vacuum) in the closure. This will include closures that are tested by mass spectrometer leakage rate testing in the gas filled envelope (hood), and evacuated envelope test methods.

The fluid flow modes taught in the typical Fluids courses are forms of viscous flow, also called continuum flow. Viscous flow modes include the laminar, turbulent, and sonic or choked flow modes. In viscous flow the molecules of a gas collide or interact primarily with other molecules of the gas.

Molecular flow occurs when the gas density (gas pressure) has been reduced, and the molecules of the gas are more likely to collide with the boundary of the conductor or container than with other gas molecules. A prediction or estimation of the flow mode can be achieved by estimating the mean free path, that is the mean distance a molecule of a gas will travel before colliding with another gas molecule. This mean free path may then be compared to the cross section of the conductance path in the closure. The mean free path is be roughly equal to $\frac{5}{Pressure in milliTorr} = the mean free path in centimeters.$

Many excellent references and articles on the topic of molecular flow may be found on the internet using the search term: *molecular flow*.

Although the cross section of the portion of the conductance path between the seals in a package closure will not typically be cylindrical, useful observations may be obtained by performing a few comparisons and illustrations from the information in Figure 1. Among these observations:

- a) The conductance and the length of the path are inversely proportional, e.g. if the length of the path is reduced by a factor of 2, then the conductance will increase by a factor of 2. This observation is not usually useful with respect to the portion of the leak test path between the seals of a closure, but it is at times useful in the design of the test port. Keeping the length of the test port path, or at least the length of the small diameter portion of the test port path short can improve the conductance of the system.
- b) The relationship between conductance and the cross section of the conductor is over-square, that is, a change in the diameter results in a change in the conductance that is more than the ratio of the squared values of the cross section.

To illustrate:

- \circ Assuming a path length of 25.4 cm (10 inches), a few estimated conductances would be:
 - 12 mm diameter conductor would have a 0.7 l/s estimated conductance
 - 6 mm diameter conductor would have a 0.15 l/s estimated conductance
 - 3 mm diameter conductor would have a 0.015 l/s estimated conductance
- Assuming a path length of 76.2 cm (30 inches), which would correspond to a closure path diameter of approximately 24 cm, a few estimated conductances would be:
 - 12 mm diameter conductor would have a 0.28 l/s estimated conductance
 - 6 mm diameter conductor would have a 0.042 l/s estimated conductance
 - 3 mm diameter conductor would have a 0.0052 l/s estimated conductance

These examples illustrate the effect of reductions in the cross section of a conductor with respect to the conductance.



Figure 1. Molecular Flow Conductance of a Cylindrical Tubing

To use Figure 1 to estimate the molecular flow conductance of a cylindrical conductor:

- 1. Find the length of the conductor along the vertical axis of the Figure. The left axis is scaled in meters, the right axis is scaled in inches.
- 2. Find the intersection of the length of path, with the curved line corresponding to the conductor diameter.
- 3. Translate the intersection of the length of path and the conductor diameter to the horizontal axis. The top of the Figure is scaled in liters per second of conductance, and the bottom axis is scaled in cubic meters per second.

PROVISION OF A SYSTEM CALIBRATION PORT IN THE PACKAGE CLOSURE DESIGN

The provision of a System Calibration Port for the package closure makes it possible for package operators to readily and robustly comply with ANSI N14.5-2014, *For Radioactive Materials – Leakage Tests on Packages for Shipment, (ANSI, 2014)* including:

- Paragraph **8.1 General** which reads in part: "...system calibrations before, and after, leakage rate measurements." (Emphasis added)
- Paragraph **8.6 Standard leaks** which reads in part: "*Standard leaks shall be used when a reference standard is necessary to perform ... system calibrations, and system response time determinations.*" (Emphasis added)

The Difference Between Instrument Calibration and System Calibration.

It is important to distinguish between an instrument calibration and a test system calibration. An instrument calibration is a calibration, or more accurately an individual instrument standardization, that is part of the preparation for a leakage rate test. Figure 2 illustrates an instrument calibration of a mass spectrometer leak detector. The instrument system calibrations for test methods other than mass spectrometer leak testing are similar in that the instrument is calibrated in isolation from the test system.

A test system calibration is a calibration of the fully assembled test system. The test system includes the test instrument, the object under test, all interconnecting hardware and software, and a calibrated standard leak that can be admitted to the test object at the conductance extremity from the test instrument connection point.



Figure 2. An Instrument Calibration, Standardization, or Verification of Calibration. (Note, the calibrated standard leak may be external, as illustrated here, or internal to the instrument.)



Figure 3. A Test System Calibration including a System Calibrated Leak Standard, Test Instrument, Test Object, and the Interconnecting Hardware.

For package closures, the system calibration port is typically designed as a duplicate of the test port that accesses the space between package closure seals. This system calibration port should be located at the extremity of the conductance path from the test port. This will usually result in placing the system calibration Port 180 degrees azimuth from the package closure test port. The photograph in Figure 4 illustrates the system calibration port at 180 degrees azimuth from the test port.

The System Calibration Port facilitates a formal test system calibration each time a leakage rate test is performed. Test system calibration is different and distinct from instrument standardization/calibration. The test system includes the test instrument, the test object (in this case the package closure under test), and all interconnecting hardware. The test system calibration typically involves the admission of a calibrated leak standard into the test system at the conductance extremity from the test instrument. (ASME) See ASME Boiler and Pressure Vessel Code Section V, Article 10, Mandatory Appendix IX, and ASTM E 1603 and other leak testing standards for a more detailed description and examples of test system calibrations, and the distinction between instrument standardization/calibration and test system calibration.

Figure 4 is a photograph of a package closure mock-up that is being leak tested by a pressure change measurement leakage rate test. The calibrated standard leak that can be admitted to the test system at the conductance extremity for the test instrument connection point is shown. This arrangement provides for the six advantages and benefits of a full system calibration of the leak test, including direct determination of the minimum test dwell time and test system calibration. In Figure 4, the test instrument is the pressure gauge.



Figure 4. A Leak Test System for a Pressure Drop Test, Including the System Calibrated Leak

Six (6) Advantages of System Calibration (with Test Port at the Extremity of the Conductance)

- Test system calibration provides six (6) specific advantages that lead to economical and reliable leak testing:
 - 1. Signal Amplitude Calibration
 - 2. System Response Time/Test Dwell Time determination
 - 3. Evidence that the test system is sensing at the time of the leakage rate measurement
 - 4. Evidence that the test system is sensing the entire package closure at the time of test
 - 5. A Test Quality Factor for test evaluation and confidence
 - 6. Procedure Qualification/Re-qualification each time a leak test is performed
 - 1. Signal Amplitude Calibration

The system calibration provides calibrated correlation of the test instrument signal to the package leakage rate. With System Calibration this correlation is measured and validated each time the leak test is performed. The test instrument signal may be a change in pressure with change in time, as occurs in pressure change leak tests, or the leakage rate signal from a mass spectrometer leak detector.

The *Calibration Factor*, as defined in ANSI N14.5-2014, Section 2.1, **Definitions**, is the signal amplitude calibration, and it is determined from the system calibration.

The inclusion of the system calibration in each test reduces uncertainty in the leakage rate test result. When the test system calibration is performed with each test, the system calibration is not dependent on a procedure qualification test that was performed at some time in the past, with a test instrument that may have been performing with a different efficiency and sensitivity than is true for the current test. A great many elements of uncertainty are eliminated by performing the system calibration in the test sequence for each leak test.

2. System Response Time/Test Dwell Time determination

The System Calibration is an empirical means of determining the required test dwell time each time a leak test is performed. Use of the system calibrated standard leak to determine the system response time is described in

(ANSI, 2014b), Paragraph 8.6, and Paragraph 2.1 Definitions

The inclusion of a system calibration that utilizes a calibrated standard leak admitted to the test system at the extremity of the leak test path, in each leak test, reduces uncertainty in the determination of the system response time and the minimum test dwell time. A properly performed system calibration empirically determines the minimum test dwell time for the specific test closure under test, with the specific test system equipment and environmental conditions existing at the time of the test.

3. Evidence that the test system is sensing at the time of the leakage rate measurement

The system calibrations provide evidence in the leak testing operation that the test system instrument is actually sensing at the time of a leakage rate measurement.

If the system calibrations are performed as part of the test sequence, and are included immediately before and after the leakage rate measurement portion of the test procedure, with no changes to the test instrument during the test sequence, then the test data includes firm evidence that the system and instrument were actually sensing at the time of the measurement.

An example may be useful here: An associate was conducting a training course in helium mass spectrometer leak testing. A demonstration of a bell jar evacuated envelope test was to be performed as part of the course. After setting up the test system for the demonstration, the students left for lunch. While the students were away, the instructor slipped an open calibrated standard leak into the bell jar to simulate a leaking test article. When the students returned following lunch, they continued with the leak test. They energized the TEST START function of the mass spectrometer leak detector (the MSLD). The lights and indicators on the MSLD cycled as expected, and indicated: NO DETECTABLE LEAK. The MSLD was cycled to VENT, then the TEST START function was again energized, and the lights and indicators on the MSLD again cycled as expected, and now correctly indicated the leakage from the calibrated leak that was hidden in the bell jar. The MSLD apparently had a logic fault, or a sticking valve. The MSLD had not previously demonstrated a fault, and worked correctly thereafter so far as we know. However, on this one occasion, it appeared to be working, but was not actually sensing for leakage. This unintended event provides a good illustration of the issue.

The System Calibration, properly performed, produces evidence in each test as to whether the test system is or is not sensing for leakage at the time of the leakage rate measurement. This adds confidence to the test operation.

4. Evidence that the test system is sensing the entire package closure at the time of the test

The admission of the system calibrated standard leak to the test system at a point that is at the extremity of the conductance path from the test instrument connection point provides evidence in each test that the test path is clear, and that the test system is sensing all the way across the package closure. Without this measure, the test operator is working from an assumption that the test system is sensing across the full closure.

A few examples may be useful:

There are verbal reports from the past which claim that there have package closures of the single test port design that have been disassembled and found to have the test path of the closure filled with vacuum grease. A leak test sequence that is performed on a closure that is packed full of vacuum grease prior to testing is unlikely to have actually leak tested the containment seal closure. The leak test instrument could not have sensed completely around the enclosure if the test path is plugged.

There is also a legacy account of a radiological site clean-up campaign that was going well. The package closures were of a single test port design. The packages were being loaded, leak tested, and staged for shipping. At some point, the test port adaptor was damaged or broken. The adaptor was a simple design, and the site personnel produced a replacement adaptor using the lathe in the machine shop. The site was able to resume leak testing, and the tests were proceeding really well. After a few packages, an inspector became

concerned. The leak testing operations were going "too well". An inspection of the replacement test adaptor revealed that the bore hole through the adaptor did not actually go all the way through the adaptor. The leak testing operation had not tested the closures at all. The test instrument was only sensing up to the end of the hole in the test adaptor.

Had the packages for these two illustrations had a system calibration port at the extremity of the leak test conductance, the leak test cycle would have faulted, and the leak test operation would have immediately stopped, forcing the leak test operators to diagnose the problem.

5. Test Quality Factor for Evaluation of Test Reliability

The Test Quality Factor (TQF) provides an objective measure of process control. The calibration factor (sensitivity factor) from the response to the system calibrated leak prior to the leakage rate measurement is divided by the calibration factor (sensitivity factor) from the response to the system calibrated leak when it is re-admitted to the test system immediately after the leakage rate measurement step. Ideally, the TQF value is 1, however, TQF values between 0.9 and 1.1 are indicative of a process that is robustly under control. TQF values between 0.77 to 1.4 are typically permitted (ASME).

The TQF is also a valuable predictive tool. Leak testing programs that are well under control typically generate values between 0.9 and 1.1. Frequently, leak testing process issues that will cause problems at a later point in time will generate progressively wider ranges of TQF values well in advance of causing production delays. Organizations that monitor the TQF values generated during testing have the opportunity to perform preventative investigation, diagnosis, and correction before production delays are encountered.

The TQF is sensitive to a wide array of variables that affect the reliability of a leak test. Subtle changes in test operations, environmental stability in the test area, cleanliness of objects under test, test instrument stability, noise in the electrical power supply, radio signal interference, and a wide array of other issues that affect test reliability have been reliably indicated through the TQF value.

The absence of a system calibration would preclude the calculation of the Test Quality Factor.

An example of the use of the Test Quality Factor can be seen in (ASME), Section V-2017, Article 10, paragraph IX-1062.6, Test Reliability – Correlation of Calibration Factors.

6. Procedure Qualification/Re-qualification each time a leak test is performed

Test procedure qualification must be considered and addressed for each leak testing procedure. Procedure qualification is one of the topics addressed in (NRC, a) 10 CFR Part 71, "Packaging and Transportation of Radioactive Material." Paragraph 71.119, **Control of special processes**, which reads in part: "...measures to assure that special processes, including ...nondestructive testing are controlled and accomplished by qualified personnel using qualified procedures in accordance with...". Test procedure qualification is also addressed in (ANSI) N14.5-2014, Paragraph 8.6., and the United States Nuclear Regulatory Commission (NRC, b), NRC INFORMATION NOTICE 2016-04, dated March 28, 2016.

Many leak testing procedures for package closures that include system calibration using a calibrated standard leak admitted to the test system at the extremity of the system test path from the instrument connection point may be deemed to be self-qualifying with each performance of the leak test. These self-qualifying test sequences include the demonstration that the test system, as used at the time of the leak test, will sense a leak of the size of the calibrated leak when that leak enters the system at the least advantageous location. This, in turn, makes it possible to change elements of the procedure that are essential variables, such as the make a model of the instrument, without having to build a qualification fixture, and without having to perform a separate documented procedure qualification effort.

If a package closure design does not include a means for admitting a calibrated standard leak at the least advantageous location, then many, or most high sensitivity leak test procedures would require fabrication of a procedure qualification fixture, and a separate procedure qualification effort with documentation. Any change in the procedure that changes an essential variable would then create a need to perform another procedure qualification test.

CROSS SECTION OF ELASTOMER SEALS

Package closures that include the use of a small cross section elastomer O-ring may be difficult to leak test by methods that require the use of a tracer gas such as helium or hydrogen/nitrogen mixtures. A wider cross section seal will typically provide a longer time interval between the introduction of the tracer gas and the arrival of the tracer gas leakage rate signal from permeation.

MATERIAL OF ELASTOMER SEALS

Some elastomer compounds for seals offer very appealing properties for the package designer. However, some of these compounds, particularly silicone based elastomers, have permeation rates that can make the closure difficult or impossible to leak test with a tracer gas such as helium or hydrogen/nitrogen mixtures

If the use of silicone seals or other compounds is absolutely unavoidable, then a very careful optimization of the other closure design variables will be necessary. Even with the optimization of the other design variables, acceptable leak testing of these closures with a tracer gas may not be possible.

PROVISION FOR THE RAPID ACHIEVEMENT OF TRACER GAS CONCENTRATION

Package designs that provide means for the rapid achievement of the tracer gas concentration for leakage rate testing provide a number of significant advantages for the package owner-user. These advantages may include:

The Ability to Perform High Sensitivity Leakage Rate Testing of the Loaded Package

The ability to perform a tracer gas leakage rate test that meets the requirements for an ANSI N14.5 "Periodic" or "Maintenance" leakage rate test of the package with a radioactive material shipment loaded in the package can be distinctly advantageous.

- This capability provides a recovery plan that does not require the unloading of the package for leak testing in the case of damage to a seal or other sealing element after the package has been loaded.
- This capability also provides a recovery plant that does not require unloading of the package, nor application for special waiver from the regulator if there is a delay in operations or shipment that cause the valid time period for the ANSI N14.5 "Periodic" leak test to expire before the package is placed with the transporter.
- This capability may substantially reduce the labor and schedule time required for periodic or maintenance tracer gas leakage rate testing of package closures. There are several package designs currently in use that require removal of the containment lid and removal of test hardware from the package cavity following completion of the tracer gas leakage rate test. In the case of large packages, this adds crane lifting of components and the related opportunity for damage of a closure. Designs that eliminate the need for this activity generate ongoing savings for the package owner-operator.

The Ability to Perform Package Closure Leak Tests in Adverse Environments - Adverse Weather.

Many, probably most, packages operated in compliance with ANSI N14.5 have the "Pre-Shipment" leakage rate test performed using a pressure change leakage rate technique. One of the challenges for gas pressure change leak testing operations is that the package closure and the area surrounding the pressure monitored test equipment must be temperature controlled, a condition described as "isothermal" in (ANSI), paragraph A5.1.4 and A.5.2.4.

If the package is designed to permit tracer gas leakage rate testing with the package loaded, then the package

owner or operator has the option to perform loaded package leakage rate tests using a tracer gas technique. This capability is particularly useful when operating in locations where the ambient temperature conditions would make pressure change leakage rate testing difficult, or impossible. Tracer gas leakage rate testing is less dependent on having a very stable temperature (i.e. "isothermal"). In extreme conditions, the tracer gas leakage rate test will be more reliable and more tolerant of temperature variation than gas pressure methods.



Three Seal Closure Designs for Rapid Achievement of Tracer Gas Concentration

Figure 5. Photo of small scale mock-ups for closure design evaluation. A common double O-ring closure to the left. Triple O-ring closure for rapid helium hood test to the right.

Figure 5 includes a photograph illustrating a three seal closure design.

The inner seal is a test seal only. It creates a small inner annulus volume between the inner seal, and the containment seal. This volume can be evacuated prior to introduction of the tracer gas. When it is time for introduction of the tracer gas, the backfill time is very short, as the volume is small, and the port into this volume is deliberately sized to allow a rapid fill. This short, rapid seal minimizes the tracer gas exposure time of the containment seal, thereby reducing the likelihood of a tracer gas permeation signal interfering with the leak test.

The outer seal is also a test seal. This is similar to the outer seal in a typical two seal package closure. When the closure is tested by a tracer gas technique, the annular volume between the outer test seal and the containment seal is evacuated and monitored by the leak testing instrument.

There is opportunity for engineering creativity in the three seal closure. We are familiar with one package design that has the inner seal as a radial seal between the package bore and a portion of the lid that fits down into the bore. This design saves diameter for the package. We are familiar with another package that places the outer seal in a radial position between a tapered lid and a tapered entrance to the package, with the containment seal and inner test seal in simple face seal positions. Again, this design saves package diameter.

One incidental benefit of the three seal design: As with two seal package closures, when the package closure is pre-shipment leakage rate tested by a gas pressure method, if there is a test failure, the test operator does not have a means of knowing if the leakage causing the test failure is a leak of the containment seal, which would be unacceptable, or a leak of the test seal, which would be acceptable. With the three seal closure, if a gas pressure test using the outer annular space fails the gas pressure test, the test operator can change over to leak test from the inner annular space. If the gas test of either annular space passes, then the containment seal is correctly installed, and the package can be shipped.

It is acknowledged here that the test ports into the inner annular space are part of the containment boundary, and must be leak tested as with any containment boundary closure.

Provision of an Adequate Vent Port

For packages with a two seal closure, the provision of a vent port to the package cavity that is sized to

support the evacuation or partial evacuation of the package cavity and rapid backfill of the cavity, or a fractional portion of the cavity, is very helpful. A small vent port into a large package cavity can cause difficulties. The tracer gas backfill may be too long to support tracer gas leakage rate testing. If the backfill of time required to achieve the helium concentration is long, then the probability of tracer gas permeation interfering with the leak test is increased.

DESIGN TO AVOID OR DEFEAT VIRTUAL LEAKS

Package closure designs that minimize the virtual leaks provide significant savings and robustness in operations. This is particularly applicable to closures that will be leakage rate tested by gas pressure means.

But what is a virtual leak? A virtual leak is a pocket that is connected by a leak path to the test volume, but that does not have a communication path to the outside of the package. By way of illustration, virtual leaks include:

- Bolts or screws that are threaded into blind holes. The pocket formed at the bottom of the hole, and the threads form a gas source to the test volume, but there is no outlet to the outside world. There are design details to defeat this, for example the use of bolts or screws that have the equivalent of a key way machined down one side of the thread, or a center drilled hole through the bolt. The keyway or hole allow the pocket at the bottom of the bolt hole to rapidly reach the same pressure as the test volume.
- Metal to metal interfaces. These may act as gas sources, and take longer to equalize in pressure with the volume of interest than would occur if the surfaces were separated by even a very small distance.
- O-ring glands. When an O-ring is compressed into the O-ring gland, a pocket of air may be trapped in the gland corner. This pocket may then leak slowly into the test volume of interest until the pocket and the test volume reach an equal gas pressure. To the gas pressure leak test, this looks like a leak. To overcome this, the leak test procedure will have a period of pressure equalization. We are aware of some larger package closures that take an hour or more for the pressure equalization to be adequate to permit a pre-shipment leakage rate test to a sensitivity of better than 1E-03 atm. cm³/sec. Figure 6 illustrates a pair of solutions that provide venting of the pocket of an O-ring gland to allow rapid pressure equalization of the pocket and the test volume of interest.



Figure 6. Examples of Two Designs for O-ring Gland Vents.

CONCLUSIONS

The design of package closures is an opportunity for creative engineering excellence. Closures that test easily can be a source of ongoing savings and benefit to the package owners and operators.

However, the balancing of the features and attributes that will result in a closure that will test easily are not

obvious, and the production of these designs is not purely analytic.

We would strongly encourage package designers to:

- 1. Take the suggestion provided in the Introduction to ANSI N14.5-2014, Appendix A, to engage: "…leak testing technology expertise early in the design process." Leak Testing is a practice, as well as an application of science. Hands-on experience with the leakage rate testing of an array of package designs is very helpful.
- 2. Perform a thorough review before copying a closure design from other packages. There are many packages in use that are very difficult to leak test. Design offices may be unaware of the level of effort and expense spent by operations personnel to leak test some existing designs.
- 3. Fabricate a mock-up of the closure design EARLY, preferably long before final design drawings are issued. The mockup should be full scale in the cross section (Test ports, O-ring cross sections and locations and test ports should all be full scale.) For large diameter packages, the mock-up may be scaled for diameter, but not for cross section. The closer to full scale for package diameter, the greater the likelihood of developing an excellent design, with fewer surprises after the design is finalized.

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