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# FIFTH INTERIM STATUS REPORT: MODEL 9975 PCV O-RING FIXTURE LONG-TERM LEAK PERFORMANCE

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**Fifth Interim Status Report: Model 9975 PCV O-Ring Fixture Long-Term Leak Performance**

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**Summary**

A series of experiments to monitor the aging performance of Viton® GLT O-rings used in the Model 9975 package has been ongoing for six years at the Savannah River National Laboratory. Sixty-seven mock-ups of 9975 Primary Containment Vessels (PCVs) were assembled and heated to temperatures ranging from 200 to 450 ºF. They were leak-tested initially and have been tested at nominal six month intervals to determine if they meet the criterion of leaktightness defined in ANSI standard N14.5-97. Fourteen additional tests were initiated in 2008 with GLT-S O-rings heated to temperatures ranging from 200 to 400 ºF.

High temperature aging continues for 36 GLT O-ring fixtures at 200 – 350 ºF. Room temperature leak test failures have been experienced in 5 of the GLT O-ring fixtures aging at 300 and 350 ºF, and in all 3 of the GLT O-ring fixtures aging at higher temperatures. No failures have yet been observed in GLT O-ring fixtures aging at 200 ºF for 30-48 months, which is still bounding to O-ring temperatures during storage in KAMS.

High temperature aging continues for 6 GLT-S O-ring fixtures at 200 – 300 ºF. Room temperature leak test failures have been experienced in all 8 of the GLT-S O-ring fixtures aging at 350 and 400 ºF. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 or 300 ºF for 19 months.

For O-ring fixtures that have failed the room temperature leak test and been disassembled, the O-rings displayed a compression set ranging from 51 – 95%. This is significantly greater than seen to date for packages inspected during KAMS field surveillance (23% average).

For GLT O-rings, service life based on the room temperature leak rate criterion is comparable to that predicted by compression stress relaxation (CSR) data at higher temperatures (350 – 400 ºF). While there are no comparable failure data yet at aging temperatures below 300 ºF, extrapolations of the data for GLT O-rings suggests the CSR model predictions provide a conservative prediction of service life relative to the leak rate criterion. Failure data at lower temperatures is needed to verify this apparent trend. Insufficient failure data exist currently to perform a similar comparison for GLT-S O-rings.

Aging and periodic leak testing will continue for the remaining fixtures.

**Background**

This is an interim status report for experiments carried out per Task Technical Plans WSRC-TR-2003-00325 [1] and SRNS-TR-2008-00054 [2], which are part of the comprehensive 9975 package surveillance program [3].

PCV test fixtures were assembled with either Parker Seals V0835-75 (hereafter referred to as Viton® GLT) O-rings or Parker Seals VM835-75 (hereafter referred to as Viton® GLT-S) O-rings, and are being aged in environments that provide varying degrees of margin over KAMS storage conditions. The purpose of these experiments is to characterize the performance of the O-ring seals, and then correlate the data to lifetime predictions of PCV and SCV O-ring seals in

9975 packages being stored in KAMS. O-ring performance in these tests is defined by leak-tightness.

The data from these fixtures are scoping in nature, although most of the controls under which they were collected are typical of baseline data. Accordingly, care should be used to assess the overall quality of the data prior to use in baseline applications. Within the 9975 surveillance program, these data will be used for information only, to compare to baseline data from other testing and build confidence in the overall predictions of O-ring seal service life.

## Experimental Method

### *Test Matrix*

Testing has evolved to include 3 test matrices. These address Viton® GLT O-rings aged at 200 or 300 ºF, Viton® GLT O-rings aged at 350 – 450 ºF, and Viton® GLT-S O-rings aged at 250 – 400 ºF.

The first test matrix was developed to determine the importance and effect of several variables on the condition of the PCV O-rings over time inside the KAMS storage facility. The variables believed to be the most relevant to O-ring performance in storage were O-ring temperature, radiation/dose rate, O-ring lubrication, and internal PCV atmosphere. Two different dose rates were selected to evaluate potential dose rate effects. A total of 62 tests, with 22 separate sets of conditions were developed. Replicates of tests were developed based on a modified full-factorial statistical design. The test variables and the basis for variable selection are given in Table 1.

The interior of the test fixture is accessible through a tube connected to the bottom. This tube includes a T connection to facilitate leak testing of both O-rings simultaneously. In May 2010, it was observed that after backfilling the designated fixtures with $CO_2$, the T side port for those fixtures was capped off, but the end of the tube was not capped. It is not known how long this practice has persisted. This oversight has negated the effectiveness of the $CO_2$ backfill as a variable since the backfill gas would not remain contained within the fixture's body. Accordingly, this parameter will no longer be credited as a valid test variable.

Several fixtures have been removed from the first test matrix since the initiation of the study. Eleven were removed from test based on leak test performance while at their conditioning temperature of 200 °F or 300 °F and they were disassembled and examined. Fourteen more were taken out of test after a power failure caused a temperature excursion severe enough to invalidate the tests. One additional fixture was removed from test in 2007 for reasons that were not documented. Fixture 62 was returned to service briefly with new O-rings in 2007, and designated 62-2007. Further details of these fixtures are provided in Reference 4. At the time of the previous status report [4], none of these fixtures had been removed from test due to failure to remain leaktight at room temperature. Subsequently, several fixtures conditioning at 300 ºF have experienced room temperature O-ring leakage. The status of each fixture, along with its test parameters, is summarized in Table 2.

Fixtures in the first test matrix are leak tested on a nominal 6-month schedule. Once the first of these began failing the room temperature leak test, the test frequency for fixtures heated to 300 °F was increased to every 3 months.

In the second test matrix, five fixtures were placed into test in October 2008 with new Viton® GLT O-rings. These fixtures were aged at temperatures ranging from 350 to 450 °F. They were intended to provide some O-ring failures in a shorter time frame to enhance the predictive value of the original test matrix and to determine the time to failure at the "continuous" service temperature rating (400 °F). The predictive model assumes that the time to leakage at all temperatures is a function of a common mechanism. With the expectation that these would fail in a much shorter time than the original fixtures, they have been leak tested on a nominal 3 week frequency. All of these fixtures were assembled with the normal O-ring lubricant and contained no backfill gas (i.e. filled with air). Three of them (one each at 350, 400 and 450 °F) were irradiated to 2E5 rad at a high dose rate.

The third test matrix repeats much of the variety of the first two matrices with Viton® GLT-S O-rings, but on a smaller scale. Seven separate sets of conditions were developed, and tested in duplicate for a total of 14 fixtures. The status of these fixtures, along with their test parameters, is summarized in Table 2.

***Initial Assembly and Setup***

The two-piece lid of the mock-up PCV, consisting of the cone seal nut and cone seal plug, was machined to be identical to the actual PCV lid. The body of the mock-up PCV was shortened to 3.5 inches from the original design of 18.6 inches and a threaded hole was machined in the bottom to provide a port for evacuating and filling the vessel with gas and for in-situ leak testing of the O-rings. A PCV test fixture with the O-rings installed in the lid is shown in Figure 1.

The mock-up PCV fixtures were assembled per the requirements described in the 9975 Safety Analysis Report for Packaging (SARP) [5]. After installation of the O-rings and assembly of the mock-up PCV test fixture, an initial leak test was performed while the fixture was at room temperature. If the fixture required irradiation, it was placed in a Co-60 gamma cell and irradiated at one of two dose rates to reach a total dose of 2E5 rad. This is equivalent to a ten year dose at the bounding dose rate expected for the PCV O-rings (2 rad/hr). The fixture was irradiated at either a "slow" dose rate of approximately 667 to 830 rad/hr or a faster rate of ~1.7E5 rad/hr. After irradiation, the fixture was leak tested again while at room temperature, and heated to test temperature.

The vessels are heated with a flexible, wound-wire heater wrapped around the vessel circumference. Ceramic fiberboard and fiber batting are used to insulate the exposed ends of the fixtures. Stainless steel tubing is attached to the port on the top of the fixture lid via a high-pressure fitting and to the hole machined into the bottom of the PCV body. A thermal fuse was added to each heater to prevent excessive temperature excursions. The heaters are controlled by a desktop computer running LabView™ software, with feedback via a type-K thermocouple attached to the PCV body. The final assembled fixture is shown in Figure 2.

Initially, there was a plan to raise the temperature of the O-rings in some of the first matrix fixtures to 400 °F for a half hour period to simulate what might occur during an accident after the O-rings had aged. 400 °F is the maximum allowable SCV/PCV O-ring temperature for short term conditions [6]. The excursions have been delayed as a result of the loss of several of the fixtures to disassembly and overheating. The objective of predicting lifetime at the specified conditioning temperatures currently takes precedence. Subsequently, estimates of O-ring temperatures in KAMS have been revised, with the result that the 300 ºF fixtures already bound a thermal scenario within the facility [7].

***Fixture Leak Testing***

The O-ring fixtures are leak-tested after initial setup, after irradiation, and periodically thereafter to the same leaktight criterion as the 9975 PCV and SCV. The outer O-rings of the 9975 PCV and SCV are credited with being leaktight while in transport and are credited with maintaining containment while in storage in the KAMS facility [5, 8].

A room temperature leakage rate of no more than 1E-7 ref·cc/sec air (2E-7 cc/sec He) demonstrates leaktightness when measured according to the requirements outlined in ANSI Standard N14.5-97 [9]. Initially, fixtures were also tested at their conditioning temperature. These additional tests were discontinued, as discussed previously [4].

Leak testing is conducted using a helium mass spectrometer leak detector of the same make and model as that currently used in annual certification testing of the 9975 PCV and SCV, although the method used has been adapted to the O-ring fixtures. A gas filled envelope test, as defined in ANSI N14.5-97 Section A.5.3 is used for the mock-up PCV fixtures. Both O-rings are tested simultaneously, with failure of either O-ring causing a failure of the test. Although this approach differs from annual certification testing, it gives results that are valid and comparable [9], and accommodates the difference in set up of the actual PCV and SCV and the mock-up PCV fixture. If a leak is found, it is possible to determine which O-ring is leaking by selectively directing the helium to either the fixture body or the closure weep hole, thus testing one O-ring at a time.

The O-ring fixture leak test program was reviewed in December 2008, prompting reconsideration of the methodology used for leak testing the mock-up PCV fixtures. One important change that was made in the conduct of the leak test involved extending the test duration until permeation of helium through the O-ring was detected [10].

Observing a permeation signal for each test provides positive evidence that the fixture and test setup are capable of transmitting a helium signal (i.e. no part of the flow path is blocked), and that helium was actually introduced into the fixture. Once a permeation signal was observed for each fixture (the permeation response is described in Reference 10), subsequent testing is conducted without the extended duration to demonstrate permeation. The history of permeation testing is summarized in Table 3. Since the GLT-S fixtures began testing in December 2008, they were tested for, and demonstrated, permeation during their baseline testing.

## Results

Mock-up PCVs have been assembled and aged in the conductance of 68 tests on GLT O-rings, and 14 tests on GLT-S O-rings. Aging parameters include conditions of temperature, irradiation, and internal gas atmosphere. This report summarizes results for these fixtures through July 1, 2010.

A total of 36 GLT O-ring fixtures and 5 GLT-S O-ring fixtures remain in test. Both O-rings have failed to remain leaktight at room temperature in 5 of the GLT O-ring fixtures, and these have been removed from service. A single O-ring has failed to remain leaktight in 5 additional GLT O-ring fixtures, and these fixtures remain in test pending failure of the second O-ring.

All of the GLT O-ring fixtures conditioning at 200 ºF have remained leaktight at room temperature, with total times at temperature ranging from 30 to 48 months (at the time of their last leak test). Six of the GLT O-ring fixtures conditioning at 300 ºF have failed to remain leaktight (in one or both O-rings) at room temperature. Failure of these was identified at exposure times ranging from 36 to 50 months. The other fixtures conditioning at 300 ºF remain in test with exposure times ranging from 30 to 48 months (at the time of their last leak test). One O-ring in one of the 2 GLT O-ring fixtures conditioning at 350 ºF failed to remain leaktight at room temperature after 10.6 months. The second 350 ºF fixture remains leaktight at room temperature after 15.4 months at temperature. All three GLT O-ring fixtures conditioning at 400 or 450 ºF have failed to remain leaktight at room temperature and have been removed from service, as reported in the previous status report [4]. The times to failure for each GLT O-ring fixture are summarized in Table 4. Detailed leak rate histories can be found in Table 5.

All of the GLT-S O-ring fixtures conditioning at 200 ºF, 250 ºF, and 300 ºF have remained leaktight at room temperature, with a total times at temperature of >19 months. Fixtures conditioned at 350 ºF failed to remain leaktight at room temperature after a period of 114 and 358 days. Fixtures conditioned at 400 ºF with no radiation exposure failed after 50-99 days. The two fixtures conditioned at 400 ºF with radiation exposure of 2E5 rad failed after 50 and 281 days. The times to failure for each GLT-S O-ring fixture are summarized in Table 4. Detailed leak rate histories can be found in Table 5.

Note that 400 °F is the upper "continuous" service temperature often quoted for O-rings of both Viton® compounds, typically based on 1000 hours (42 days) exposure [11]. However, seal manufacturers advise that such ratings are generic only and that every design is different. End-users are therefore encouraged to perform specific tests to determine actual service limits.

The two GLT O-ring fixtures (31 and 32) conditioning at 300 °F in which both O-rings failed were disassembled in May 2010. The following observations were made during disassembly:

- Both fixtures had heavy accumulation of grease on and around the closure nut (Figure 3).
- The inner O-ring in fixture 32 was stuck tight in the plug groove.
- The body and/or plug retained black deposits from the O-rings to varying degrees in each fixture (Figure 4).

- A brown ring was observed around most of the O-ring ID and OD surfaces for fixture 31, especially the outer O-ring (Figure 3). This is indicative of oxidized grease.
- Both O-rings from fixture 32 had material missing and/or cracks. The inner O-ring had a linear indication ~1 inch long on the ID surface. It has the appearance of a shallow crack, but might reflect the loss of material that stuck to the fixture. The outer O-ring had much more distinct linear regions of material loss along the ID surface, with several small radial cracks as well. These were clustered in several locations (Figure 5).

Observations made during disassembly of the 8 GLT-S fixtures (with both O-rings failed) include:

- Fixture 38H: Metal very clean, no residue. O-rings difficult to remove from plug. Outer ring- Missing material, and/or cracks on mold line, one pin-hole, but not through wall (Figure 6).
- Fixture 39H: Bits of black dust in threads. O-rings look smooth, but flush with metallic plug. Grease looks uniform. Some oxidized grease on side flattened due to compression set, however, still elastic when pulled.
- Fixture 45H: Slight white oxidized grease. Large black band on inside of metallic body.
- Fixture 50H: No comments.
- Fixture 58H: Very easy to remove. Drops of grease present. No white oxidized grease. Remained elastic. No black band on inside of body.
- Fixture 60H: O-rings look dry while on cone seal of plug. Moderate adhesion to metallic body.
- Fixture 28H: Light brown band inside body. O-ring easy to remove, no white oxidation.
- Fixture 50H: Brown residue on plug and inside body. Purple residue on side of plug with O-ring. O-ring difficult to remove from cone seal plug.

All of the O-rings removed during these examinations received dimensional and hardness measurements. Average hardness readings for each O-ring varied from 75 to 80 Durometer M. The hardness of new O-rings is specified as 75 +/- 5 Durometer A. While there is no exact conversion between the A and M scales, they are generally very similar in value. The hardness increased slightly from baseline measurements for one fixture, and decreased slightly for the other. These differences are not considered significant.

The dimensional measurements were used to calculate compression set. The measurements were taken at different times following removal of the O-rings, but generally fall into 2 time intervals. Measurements were taken either within 4 hours of removal, or at least 9 days after removal, with some O-rings receiving multiple measurements in one or both time intervals. Most of the relaxation that was seen occurred prior to the first longer-term measurement (around 10 days). Compression set results are summarized in Table 6.

Fixture 34 was removed from service in 2007 after an inadvertent temperature excursion. The maximum temperature recorded for this fixture was 721 ºF. With temperature data being recorded every 10 minutes, the actual maximum temperature was likely higher than this value. At the time this fixture was opened, it was observed that portions of the O-rings had extruded from their grooves in the cone seal plug. Fourier Transform Infra Red (FTIR) spectroscopic analysis of the O-ring material showed that it was still consistent with Viton® GLT. More recently, another test has been performed on the outer O-ring from this fixture [12]. Dynamic Mechanical Analysis (DMA) testing measures the mechanical response of the O-ring over a range of temperatures. In this case, the testing was performed over the range -60 to +20 ºC to identify the glass transition temperature. Similar samples from a non-aged O-ring and an O-ring aged at ~265 ºF for 461 days were tested for comparison. The glass transition temperature for each of these three samples was in close agreement, indicating that degradation of the chemical structure in the Fixture 34 O-ring was minimal, at least with respect to thermal transitions.

## Discussion

Each fixture has been tested for permeation. This involves continuing the leak test until the leak rate begins to increase at a continually increasing rate, and is distinguished from a leaking fixture by a time lag between test initiation and the beginning of permeation. In cases where no permeation was seen, subsequent leak tests repeated the effort to show permeation. Permeation results are summarized in Table 3. Permeation has been seen in all but 1 fixture (fixture 5) to date. There is considerable variation in the time to permeate among the fixtures, and among multiple trials for the same fixture. Some tests permeated in as little as 5 minutes, while other tests showed no permeation for 60 minutes. There is no apparent trend in this variation. Reasons for the variation are unknown, although limited testing has shown that the amount of vacuum grease on the O-ring can affect permeation performance [10].

Extending the leak test to demonstrate a permeation signal provides two functions; it demonstrates an open flow path from the fixture to the detector, and that helium was actually introduced to the fixture. This reduced the chance for false positive results (i.e. leaktight for reasons other than seal performance). Based on the permeation results to date, an open flow path has been demonstrated for all but 1 fixture. This fixture will continue to be tested for permeation in the future until it shows permeation or until it fails. If it fails before showing permeation, its validity will be assessed based on all available data. The remaining fixtures have shown an open flow path for permeation. In general, they will not be tested to permeation in future leak tests. The test method provides sufficient assurance that helium is provided to each fixture since the bag around the fixture is seen to inflate with helium after it is evacuated.

Compression set values for the O-ring fixtures have been calculated as follows, per ASTM D395 Method B:

$$\text{Comp Set} = -(\text{initial } t_r - \text{final } t_r) / (\text{initial } t_r - \text{average O-ring groove depth}) * 100$$

with $t_r$ = radial O-ring thickness

For the GLT and GLT-S O-ring fixtures that were examined following failure of a room temperature leak test, the compression set ranges from 51 to 95%, with an average value of 83%, for measurements within the first 30 minutes. The average compression set was higher for the GLT-S O-rings (89%) than for the GLT O-rings (74%). There is a difference between the compression set for GLT O-rings aged at 300 ºF (84% avg) and GLT O-rings aged at 400 – 450 ºF (68% avg). There is insufficient range in temperature for failed GLT-S O-rings to identify whether a similar trend exists for that compound. However, the GLT-S O-rings exhibit the same compression set at 350 and 400 ºF (89% avg).

In comparison, the average compression set measured on O-rings during field surveillance to date is 23%, following storage periods in KAMS of up to 7 years. This is significantly less than measured on any O-ring (GLT or GLT-S) which was aged to the point that it did not pass a room temperature leak test.

Some of the compression set values cited above and in Table 6 differ from that cited in previous documents. Some compression set values cited in the past were calculated per ASTM D395 Method A, which does not incorporate the effect of the O-ring groove depth. All values cited in this report are based on Method B, which does incorporate this effect. For the 9975 O-ring configuration, Method B is the preferred approach since it yields compression set values that range from 0 to 100%, while Method A would yield values over a smaller range.

A separate task [2, 13] is underway to correlate the compression stress relaxation (CSR) of O-rings with aging temperature. While CSR does not provide a direct correlation to leak tightness, it is hoped that the time for the compressive force to relax to a specific value (such as 90% or 100% loss of initial force) may provide a degree of correlation with the service life based on leak-tightness. Lifetime estimates for GLT O-rings based on CSR data have been presented in Reference 14. With several room temperature leak failures of aged O-ring fixtures, an initial comparison is now possible to demonstrate whether CSR data correlates with leak data. This comparison is shown in Figure 7 for GLT O-rings. Lifetime estimates based on the two parameters are similar at higher temperatures (~350 – 400 ºF). Figure 7 also suggests that the CSR data provide a conservative life prediction at lower temperatures with regards to the actual time to leak failure of the O-rings. This observation should be viewed with caution, however, since the lower temperature portion of both curves is based on extrapolation beyond the available data. Insufficient data currently exist to draw a similar comparison for GLT-S O-rings.

## **<u>Conclusions</u>**

High temperature aging continues for 36 GLT O-ring fixtures at 200 – 350 ºF, and for 6 GLT-S O-ring fixtures at 200 – 300 ºF. Room temperature leak test failures have been experienced in 5 of the GLT O-ring fixtures aging at 300 and 350 ºF, and in all 3 of the GLT O-ring fixtures aging at higher temperatures. No failures have yet been observed in GLT O-ring fixtures aging at 200 F for 30-48 months, which is more representative of (but still bounding to) O-ring temperatures during storage in KAMS.

High temperature aging continues for 6 GLT-S O-ring fixtures at 200 – 300 ºF. Room temperature leak test failures have been experienced in all 8 of the GLT-S O-ring fixtures aging at 350 and 400 ºF. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 or 300 ºF for 19 months.

For GLT O-ring fixtures that have failed the room temperature leak test and been disassembled, the O-rings displayed a compression set ranging from 51 – 95%. GLT-S O-rings that have been disassembled displayed compression set values of 78 – 95%. These ranges are significantly greater than seen to date for packages inspected during KAMS field surveillance (23% average).

For GLT O-rings at higher temperatures (350 – 400 ºF), service life based on the room temperature leak rate criterion is comparable to that predicted by compression stress relaxation (CSR) data. While there are no comparable failure data yet at aging temperatures below 300 ºF, extrapolations of the data for GLT O-rings suggests the CSR model predictions provide a conservative prediction of service life relative to the leak rate criterion at KAMS storage conditions. Failure data at lower temperatures are needed to verify this apparent trend. Insufficient failure data exist currently to perform a similar comparison for GLT-S O-rings.

Aging and periodic leak testing will continue for the remaining fixtures.

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Table 1. Test Matrix Variables for O-Ring Experiment

| Test Variable | Values Tested | Basis for Values Tested |
|---|---|---|
| Temperature | 200 °F (93 °C) | With loss of ventilation in the KAMS facility, the maximum ambient temperature is 137 °F [15], and the corresponding PCV O-ring temperature is 199 °F [16]. |
| | 300 °F (149 °C) | The maximum allowable temperature for the PCV O-rings for continuous operation is 300 °F [5]. |
| | 350, 400, 450 °F (177, 204, 232 °C) | Elevated temperatures added to increase the likelihood of seeing O-ring failures in shorter test periods. |
| Radiation Dose | 2E5 Rad in 72 min | The bounding dose rate for the PCV is 2 rad/hr. A total dose of 2E5 rad represents ten years of storage. |
| | 2E5 Rad in >200hr | Longer-term exposure may reveal the added effect of diffusion-limited oxidation (DLO) that only occurs with long-term exposure. |
| | None | Many packages will have little radiation exposure. This also serves as an experimental control. |
| Internal Atmosphere | 75% $CO_2$ with a balance of Air * | The free volume of the PCV is filled/diluted with $CO_2$ as a cover gas. A small portion of the air originally in the vessels may remain. |
| | Air | It supplies comparative data and acts as a control. |
| O-Ring Lubrication | Silicone high-vacuum grease | It is specified in assembly of the 9975 package [3]. |
| | Krytox® 240AC | It has been used on 9975 O-rings at DOE facilities. It is used on lid components of the 9975 PCV and SCV [3]. |
| | None | It supplies comparative control data. Also, it is possible that the O-rings may be mistakenly installed without grease. |

* Recent observations identified that the practice for backfilling fixtures with CO2 did not consistently include capping off one access point after backfilling. Accordingly, this parameter has not been effectively achieved, and it cannot be credited as a variable in the test matrix.

Table 2. Summary of test parameters for fixtures

| Temp. °F | Gamma Dose (rad) / Dose Rate | Lubricant | Fixtures Still in Test | Fixtures Removed from Test | |
|---|---|---|---|---|---|
| | | | | Failed Leak Test at Room Temp | For Other Reasons |
| GLT O-ring Fixtures – First Test Matrix | | | | | |
| 200 | ~2E5 High | Normal | 5, 6, 9, 27, 36, 37, 40, 41, 42, 53, 54, 55 | | 15, 16, 23, 24 |
| 200 | ~2E5 Low | Normal | 10, 11 | | |
| 200 | No | Normal | 1, 3, 43, 44, 56, 57 | | 13, 28, 29 |
| 300 | ~2E5 High | Normal | 7, 8, [12], 26, 51, 52 | 31 | 17, 22, 25, 39, 45, 46, 47, 58, 59, 60 |
| 300 | ~2E5 Low | Normal | 18, [30] | 32 | 21, 38 |
| 300 | No | Normal | 4, 33, 49, [61], | | 2, 14, 48, 50, 62 |
| 300 | ~2E5 High | None | | | 19 |
| 300 | No | None | | | 34 |
| 200 | No | Krytox | 35 | | |
| 300 | ~2E5 Low | Krytox | 20 | | |
| GLT O-ring Fixtures – Second Test Matrix | | | | | |
| 350 | ~2E5 High | Normal | 18D | | |
| 350 | No | Normal | 19D | | |
| 400 | ~2E5 High | Normal | | 14D | |
| 400 | No | Normal | | 21D | |
| 450 | ~2E5 High | Normal | | 23D | |
| GLT-S O-ring Fixtures – Third Test Matrix | | | | | |
| 200 | None | Normal | 13H, 15H | | |
| 250 | None | Normal | 22H, 16H | | |
| 300 | None | Normal | 29H, 34H | | |
| 350 | None | Normal | | 38H, 39H | |
| 400 | None | Normal | | 45H, 58H | |
| 400 | None | Normal | | 60H, 62H | |
| 400 | 2E5 in 72 min. | Normal | | 28H, 50H | |

Table 3. Summary of testing GLT fixtures for permeation signal. Testing for permeation was repeated only if it was not seen on previous tests. Tests were typically terminated after 30 minutes, although a few tests continued for longer periods.

| Fixture Number | # Tests before Permeation Seen | Permeation Seen? | Time to Permeate |
|---|---|---|---|
| 1 | 1 | Y | 10 min |
| 3 | 1 | Y | 30 min |
| 4 | 5 | Y | 61 min |
| 5 | 3 | N (up to 30 min.) | |
| 6 | 2 | Y | 5 min |
| 7 | 1 | Y | 10 min |
| 8 | 1 | Y | 16 min |
| 9 | 1 | Y | 7 min |
| 10 | 2 | Y | 11 min |
| 11 | 1 | Y | 10 min |
| 12 | 2 | Y | 35 min |
| 18 | 1 | Y | 11 min |
| 20 | 1 | Y | 30 min |
| 26 | 1 | Y | 9 min |
| 27 | 2 | Y | 8 min |
| 30 | 1 | Y | 8 min |
| 31 | 1 | Y | 10 min |
| 32 | 1 | Y | 13 min |
| 33 | 2 | Y | 9 min |
| 35 | 3 | Y | 12 min |
| 36 | 3 | Y | 6 min |
| 37 | 3 | Y | 18 min |
| 40 | 2 | Y | 14 min |
| 41 | 1 | Y | 10 min |
| 42 | 1 | Y | 8 min |
| 43 | 3 | Y | 30 min |
| 44 | 1 | Y | 12 min |
| 49 | 1 | Y | 7 min |
| 51 | 2 | Y | 4 min |
| 52 | 2 | Y | 7 min |
| 53 | 1 | Y | 30 min |
| 54 | 1 | Y | 12 min |
| 55 | 1 | Y | 11 min |
| 56 | 1 | Y | 10 min |
| 57 | 1 | Y | 13 min |
| 61 | 3 | Y | 20 min |

Table 4. Summary of GLT and GLT-S O-ring leak failures

| | | Days at temperature before failure | |
|---|---|---|---|
| Fixture | Temp (ºF) | Inner | Outer |
| GLT O-ring Fixtures | | | |
| 26 | 300 | N A | 1273 |
| 31 | 300 | 1291 | 1291 |
| 32 | 300 | 1352 | 1352 |
| 49 | 300 | 1276 | N A |
| 52 | 300 | N A | 1097 |
| 61 | 300 | N A | 1252 |
| 18D | 350 | N A | 324 |
| 14D | 400 | 45 | 45 |
| 21D | 400 | 28 | 28 |
| 23D | 450 | 12 | 8 |
| GLT-S O-ring Fixtures | | | |
| 38H | 350 | 358 | 358 |
| 39H | 350 | 114 | 114 |
| 45H | 400 | 99 | 99 |
| 58H | 400 | 75 | 75 |
| 60H | 400 | 50 | 50 |
| 62H | 400 | 50 | 50 |
| 28H | 400 | 50 | 50 |
| 50H | 400 | 281 | 281 |

Table 5. Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 1 | |
|---|---|
| 200 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.3 E-9 |
| 6 | <9.0 E-10 |
| 18 | 8.0 E-9 |
| 30 | <1.2 E-8 |
| 36 | <1.2 E-8 |
| 42 | <1.2 E-8 |
| 48 | <2.0 E-8 |
| | |

| Test 3 | |
|---|---|
| 200 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <2.9 E-9 |
| 6 | 1.0 E-7 |
| 15 | 2.7 E-8 |
| 18 | <1.2 E-8 |
| 31 | <1.3 E-8 |
| 37 | <2.0 E-9 |
| 42 | 4.0 E-9 |
| 48 | 2.5 E-8 |

| Test 4 | |
|---|---|
| 300 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.8 E-9 |
| 6 | 1.6 E-8 |
| 12 | 2.5 E-8 |
| 19 | <8.0 E-9 |
| 32 | <1.0 E-8 |
| 38 | <8.0 E-9 |
| 42 | <1.4 E-8 |
| 48 | <3.0 E-8 |

| Test 5 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <2.3 E-9 |
| 6 | 1.1E-7 |
| 11 | 2.0 E-8 |
| 18 | <1.2 E-8 |
| 31 | <1.0 E-8 |
| 37 | <6.0 E-9 |
| 42 | <1.0 E-8 |
| 48 | <1.2 E-8 |
| | |

| Test 6 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 3.3 E-9 |
| 6 | 4.0 E-8 |
| 11 | 5.5 E-8 |
| 18 | <1.2 E-8 |
| 31 | <1.8 E-8 |
| 37 | <2.2 E-8 |
| 42 | <1.2 E-8 |
| 48 | <2.8 E-8 |
| | |

| Test 7 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 4.2 E-8 |
| 6 | 8.6 E-8 |
| 14 | <1.1 E-8 |
| 17 | <1.0 E-8 |
| 33 | <1.2 E-8 |
| 37 | 2.4 E-9 |
| 42 | <9.7 E-10 |
| 44 | <2.0 E-8 |
| 48 | <1.3 E-8 |

| Test 8 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 2.0 E-8 |
| 6 | 1.1 E-7 |
| 12 | <1.1 E-8 |
| 18 | <1.0 E-8 |
| 37 | <1.2 E-8 |
| 42 | 4.4 E-9 |
| 47 | <1.6 E-8 |
| | |

| Test 9 | |
|---|---|
| 200 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 5.6 E-9 |
| 6 | 9.9 E-8 |
| 12 | 4.2 E-8 |
| 18 | <1.8 E-8 |
| 28 | <1.6 E-8 |
| 36 | <9.5 E-10 |
| 40 | 1.0 E-7 |
| 42 | <1.0E-8 |

| Test 10 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /240 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 7.4 E-8 |
| 6 | 1.2 E-7 |
| 13 | <1.2 E-8 |
| 26 | 5.6 E-9 |
| 33 | <8.0 E-9 |
| 36 | 2.6 E-9 |
| 42 | 5.4 E-8 |
| | |

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 11 | |
|---|---|
| 200 ºF, Air fill, 1.4E5 Rad /479 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.5 E-9 |
| 8 | <1.3 E-8 |
| 14 | <8.8 E-9 |
| 25 | <1.0 E-8 |
| 31 | <4.0 E-9 |
| 35 | 4.0 E-9 |
| 42 | <1.8 E-8 |

| Test 12 | |
|---|---|
| 300 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.3 E-8 |
| 5 | 2.0E-8 |
| 9 | <1.4 E-8 |
| 20 | <1.4 E-8 |
| 24 | 1.1 E-8 |
| 30 | 8.7 E-9 |
| | |

| Test 18 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /246 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.5 E-9 |
| 7 | <1.1 E-8 |
| 10 | <6.0 E-9 |
| 26 | <1.8 E-8 |
| 29 | 2.6 E-9 |
| 36 | 6.2 E-8 |
| 39 | * |

* Test 18 – Overall fixture test at 39 months not successful, but each O-ring passed individually

| Test 20 | |
|---|---|
| 300 ºF, Air fill, 1.75E5 Rad /562 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.2 E-8 |
| 6 | 3.0 E-8 |
| 13 | <4.8 E-8 |
| 27 | <1.8 E-8 |
| 33 | <6.0 E-9 |
| 42 | <7.2 E-10 |
| 45 | <2.0 E-8 |

| Test 26 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.8 E-8 |
| 12 | <1.0 E-8 |
| 29 | <2.0 E-8 |
| 36 | <1.0 E-8 |
| 41 | <2.0 E-8 |
| 42 | >9.0 E-5 * |

| Test 27 | |
|---|---|
| 200 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 1.6 E-9 |
| 6 | 1.5 E-8 |
| 12 | <1.0 E-8 |
| 19 | <3.2 E-8 |
| 24 | <1.2 E-8 |
| 30 | <1.4 E-8 |
| | |

* Test 26 – outer O-ring failed at 42 months

| Test 30 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /300 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 5.6 E-8 |
| 6 | 1.5 E-8 |
| 11 | <1.8 E-8 |
| 24 | <2.0 E-8 |
| 32 | <3.0 E-8 |
| 36 | 5.3 E-9 |
| 42 | 2.1 E-9 |
| | |

| Test 31 | |
|---|---|
| 300 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.5 E-8 |
| 11 | <1.2 E-8 |
| 24 | <1.6 E-8 |
| 35 | 2.6 E-8 |
| 42 | <2.6 E-8 |
| 45 | 8.2 E-7 * |
| | |

| Test 32 | |
|---|---|
| 300 ºF, Air fill, 2E5Rad /300 hr | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.3 E-9 |
| 6 | 5.9 E-8 |
| 11 | <8.0 E-9 |
| 25 | <3.0 E-8 |
| 32 | <1.8 E-8 |
| 36 | 2.6 E-8 |
| 42 | <1.4 E-8 |
| 44 | 2.9 E-6 ** |

* Test 31 – both O-rings failed at 42 months

** Test 32 – both O-rings failed at 44 months

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 33 | |
|---|---|
| 300 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.3 E-8 |
| 10 | <2.0 E-8 |
| 22 | <1.8 E-8 |
| 29 | <2.6 E-8 |
| 36 | <1.0 E-9 |
| 38 | <3.2 E-8 |
| 42 | * |

| Test 35 | |
|---|---|
| 200 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.3 E-8 |
| 11 | <1.4 E-8 |
| 24 | <1.6 E-8 |
| 31 | <3.2 E-8 |
| 36 | <1.2 E-8 |
| 42 | <1.6 E-8 |
| | |

| Test 36 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <1.5 E-9 |
| 6 | 1.3 E-8 |
| 11 | <8.0 E-9 |
| 25 | <1.8 E-8 |
| 32 | <2.8 E-8 |
| 36 | <1.0 E-8 |
| 42 | <2.0 E-8 |
| | |

* Test 33 – Overall fixture test at 42 months not successful, but each O-ring passed individually

| Test 37 | |
|---|---|
| 200 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 8 | 1.9E-8 |
| 11 | <8.0E-9 |
| 24 | <1.8E-8 |
| 32 | <2.2E-8 |
| 36 | <1.0E-8 |
| 41 | <3.0 E-8 |

| Test 40 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 7.7E-9 |
| 6 | 1.5 E-8 |
| 12 | <1.0 E-8 |
| 28 | <2.6 E-8 |
| 35 | <7.0 E-10 |
| 42 | 1.6E-8 |
| | |

| Test 41 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.3 E-8 |
| 11 | <1.0 E-8 |
| 24 | <1.9 E-8 |
| 32 | <1.8 E-8 |
| 36 | 2.0 E-9 |
| 42 | <2.0E-8 |

| Test 42 | |
|---|---|
| 200 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.5 E-8 |
| 11 | <1.0 E-8 |
| 24 | <1.8 E-8 |
| 32 | <1.8 E-8 |
| 36 | 1.8 E-9 |
| 42 | <2.2 E-8 |

| Test 43 | |
|---|---|
| 200 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 2.0 E-8 |
| 12 | <1.0 E-8 |
| 24 | <1.8 E-8 |
| 33 | <3.0 E-8 |
| 35 | 1.8 E-9 |
| 42 | <5.8 E-8 |

| Test 44 | |
|---|---|
| 200 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.5 E-8 |
| 12 | <1.6 E-8 |
| 24 | <1.8 E-8 |
| 32 | <2.0 E-8 |
| 36 | 1.8 E-9 |
| 42 | 1.1 E-7 |

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 49 | |
|---|---|
| 300 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.6 E-8 |
| 11 | <6.0 E-9 |
| 30 | <1.8 E-8 |
| 36 | <1.4 E-8 |
| 42 | 1.7 E-7 * |
| 44 | 6 E-8 |
| | |

| Test 51 | |
|---|---|
| 300 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 4.3 E-8 |
| 6 | 5.2 E-8 |
| 11 | <8.0 E-9 |
| 21 | <2.0 E-9 |
| 28 | <3.2 E-8 |
| 36 | <6.7 E-10 |
| 39 | <1.6 E-8 |
| | |

| Test 52 | |
|---|---|
| 300 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.8 E-8 |
| 11 | <1.0 E-8 |
| 21 | <2.4 E-8 |
| 27 | <2.8 E-8 |
| 32 | <1.0 E-8 |
| 36 | <2.6 E-8 |
| 36 | 4.3 E-7 ** |

* Test 49 – inner O-ring failed at 42 months. Subsequent results on outer O-ring only
** Test 52 – outer O-ring failed at 36 months. Subsequent results on inner O-ring only

| Test 53 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.8 E-8 |
| 11 | <1.0 E-8 |
| 25 | <1.8 E-8 |
| 32 | <1.8 E-8 |
| 36 | 1.8 E-9 |
| 42 | <1.2 E-8 |

| Test 54 | |
|---|---|
| 200 ºF, $CO_2$ fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.8E-8 |
| 11 | <1.2E-8 |
| 25 | <1.8E-8 |
| 32 | <1.2E-8 |
| 36 | 2.0E-9 |
| 42 | <2.0 E-8 |

| Test 55 | |
|---|---|
| 200 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.6 E-8 |
| 11 | <1.4 E-8 |
| 24 | <1.6 E-8 |
| 32 | <8.0 E-9 |
| 36 | 1.1E-8 |
| 42 | 3.7 E-8 |

| Test 56 | |
|---|---|
| 200 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.3 E-8 |
| 11 | <1.8 E-8 |
| 23 | <1.8 E-8 |
| 30 | <1.0 E-8 |
| 36 | <1.2 E-8 |
| 42 | <2.4E-8 |

| Test 57 | |
|---|---|
| 200 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.6 E-8 |
| 11 | <1.2 E-8 |
| 23 | <2.4 E-8 |
| 31 | <1.2 E-8 |
| 33 | <1.0 E-8 |
| 42 | <4.4 E-8 |

| Test 61 | |
|---|---|
| 300 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | (invalid) |
| 6 | 1.8 E-8 |
| 11 | <1.0 E-8 |
| 23 | <1.6 E-8 |
| 30 | <2.0 E-8 |
| 36 | 1.8 E-8 |
| 42 | 4.7 E-8 |

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 18D | |
|---|---|
| 350 ºF, Air fill, 2E5Rad /72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | <8.0 E-9 |
| 0.3 | 1.6 E-8 |
| 0.9 | 3.2 E-8 |
| 1.5 | <8.1 E-9 |
| 2.2 | <1.9 E-9 |
| 3.0 | 5.0 E-8 |
| 3.5 | 6.1 E-9 |
| 4.2 | 9.9 E-10 |
| 4.7 | 3.6 E-9 |
| 5.4 | 1.9 E-8 |
| 5.9 | 1.3 E-9 |
| 6.4 | 2.0 E-8 |
| 7.1 | 3.3 E-9 |
| 7.6 | <6.0 E-9 |
| 8.0 | <6.0 E-9 |
| 8.5 | <9.8 E-10 |
| 8.9 | <1.8 E-8 |
| 9.5 | <8.8 E-10 |
| 10.0 | <7.4 E-10 |
| 10.6 | 6.0 E-5 ** |
| 11.0 | 3.8 E-8 |
| 11.6 | <4.6 E-8 |
| 12.3 | <2.8 E-8 |
| 12.6 | <5.8 E-8 |
| 13.2 | 8.9 E-9 |
| 13.9 | 9.4 E-8 |
| 14.5 | 1.8E-8 |

| Test 19D | |
|---|---|
| 350 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 2.8 E-8 |
| 0.3 | <7.9 E-9 |
| 0.9 | 2.3 E-8 |
| 1.5 | <8.6 E-9 |
| 2.2 | <1.9 E-9 |
| 3.0 | 2.2 E-8 |
| 3.5 | <2.8 E-9 |
| 4.2 | 1.4 E-9 |
| 4.7 | 2.0 E-8 |
| 5.3 | 7.6 E-9 |
| 5.9 | 1.5 E-9 |
| 6.4 | 4.8 E-9 |
| 7.1 | <8.0 E-9 |
| 7.6 | <1.0 E-8 |
| 8.0 | <1.0 E-8 |
| 8.5 | <1.0 E-9 |
| 9.0 | <2.2 E-8 |
| 9.6 | <8.8 E-10 |
| 10.1 | <7.0 E-10 |
| 10.8 | 3.4 E-8 |
| 11.5 | <1.2 E-8 |
| 14.8 | 5.9 E-9 |
| 15.4 | 1.3 E-8 |
| 16.0 | 1.5E-8 |
| | |
| | |
| | |

** Test 18D – outer O-ring failed at 10.6 months. Subsequent results on inner O-ring only.

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 13H (GLT-S) | |
|---|---|
| 200 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 8.5E-09 |
| 0.5 | 1.1E-08 |
| 1 | 2.3E-08 |
| 2 | 9.5E-09 |
| 2 | 1.0E-08 |
| 2 | 2.3E-09 |
| 2 | 1.1E-09 |
| 3 | 1.5E-09 |
| 3 | 4.4E-09 |
| 4 | 8.0E-09 |
| 5 | 2.8E-09 |
| 5 | 8.0E-09 |
| 6 | 1.8E-08 |
| 7 | 8.9E-10 |
| 7 | 7.16E-10 |
| 8 | 1.8E-08 |
| 8 | 1.4E-08 |
| 9 | 1.0E-08 |
| | |
| | |

| Test 15H (GLT-S) | |
|---|---|
| 200 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.8E-09 |
| 0.5 | 1.0E-08 |
| 1 | 3.1E-08 |
| 2 | 3.6E-08 |
| 2 | 2.7E-09 |
| 3 | 1.1E-09 |
| 3 | 1.2E-09 |
| 4 | 1.1E-09 |
| 4 | 2.9E-09 |
| 5 | 4.0E-09 |
| 6 | 3.5E-09 |
| 7 | 9.12E-10 |
| 7 | 2.4E-08 |
| 8 | 1.0E-09 |
| 8 | 7.29E-10 |
| 9 | 1.8E-08 |
| 9 | 1.4E-08 |
| 14 | 1.5E-08 |
| | |
| | |

| Test 16H (GLT-S) | |
|---|---|
| 250 ºF, Air fill, No rad.. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.1E-09 |
| 1 | 1.1E-08 |
| 1 | 4.4E-07 |
| 1 | 8.1E-09 |
| 2 | 9.0E-10 |
| 2 | 1.8E-09 |
| 3 | 1.2E-09 |
| 3 | 1.5E-09 |
| 4 | 6.6E-09 |
| 4 | 1.2E-08 |
| 5 | 8.0E-09 |
| 5 | 8.0E-09 |
| 6 | 8.85E-10 |
| 6 | 1.6E-08 |
| 7 | 8.63E-10 |
| 7 | 7.2E-10 |
| 8 | 1.8E-08 |
| 9 | 1.5E-08 |
| 10 | 1.6E-08 |
| 11 | 1.5E-07 |

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 22H (GLT-S) | |
|---|---|
| 250 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.1E-09 |
| 0.5 | 1.0E-08 |
| 1 | 9.0E-09 |
| 1 | 2.9E-09 |
| 2 | 2.0E-09 |
| 2 | 2.6E-09 |
| 3 | 1.0E-09 |
| 4 | 3.7E-09 |
| 4 | 8.0E-09 |
| 5 | 9.627E-10 |
| 5 | 8.0E-09 |
| 6 | 9.12E-10 |
| 6 | 2.8E-08 |
| 7 | 1.09E-09 |
| 7 | 7.29E-10 |
| 8 | 2.0E-08 |
| 9 | 2.6E-08 |
| 9 | 1.8E-08 |
| 11 | 2.0E-08 |
| 12 | 3.9E-08 |
| | |
| | |

| Test 29H (GLT-S) | |
|---|---|
| 300 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 8.2E-09 |
| 0.5 | 1.1E-08 |
| 0.5 | 1.6E-08 |
| 1 | 8.1E-09 |
| 2 | 6.2E-09 |
| 2 | 1.0E-09 |
| 3 | 1.5E-09 |
| 3 | 1.1E-09 |
| 4 | 2.2E-08 |
| 4 | 4.2E-09 |
| 5 | 9.9E-09 |
| 5 | 9.627E-10 |
| 6 | 8.0E-09 |
| 6 | 9.12E-10 |
| 6 | 2.4E-08 |
| 7 | 1.07E-09 |
| 7 | 6.74E-10 |
| 8 | 1.8E-08 |
| 9 | 1.2E-08 |
| 10 | 2.0E-08 |
| 11 | 2.9E-08 |
| 12 | 1.0E-08 |

| Test 34H (GLT-S) | |
|---|---|
| 300 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.5E-09 |
| 1 | 1.0E-08 |
| 1 | 2.0E-09 |
| 2 | 8.0E-09 |
| 2 | 3.2E-09 |
| 3 | 1.0E-09 |
| 3 | 2.2E-09 |
| 4 | 1.1E-09 |
| 4 | 8.8E-10 |
| 5 | 2.4E-09 |
| 5 | 1.0E-08 |
| 6 | 9.502E-09 |
| 7 | 1.2E-08 |
| 7 | 9.12E-10 |
| 8 | 2.4E-08 |
| 8 | 1.05E-09 |
| 9 | 6.8E-09 |
| 9 | 1.6E-08 |
| 10 | 1.6E-08 |
| 11 | 1.6E-08 |
| 12 | 3.0E-08 |
| 13 | 1.16E-09 |

| Test 28H (GLT-S) | |
|---|---|
| 400 ºF, $CO_2$ fill, 2E05 in 72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.1E-9 |
| 0 | 1.1E-8 |
| 0.5 | 1.9E-9 |
| 0.8 | 4.9E-8 |
| 1.6 | FAILED |
| | |
| | |
| | |
| | |

| Test 45H | |
|---|---|
| 400 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.3E-9 |
| 0 | 1.0E-8 |
| 0.5 | 1.9E-9 |
| 0.8 | Outer Failed |
| 1.6 | 3.4E-6* |
| 2.2 | 1.0E-9* |
| 2.4 | 2.1E-9* |
| 2.6 | 1.1E-9* |
| 3.3 | FAILED |

| Test 58H | |
|---|---|
| 400 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 1.5E-8 |
| 0 | 1.0E-8 |
| 0.5 | 5.1E-8 |
| 0.8 | 7.8E-9 |
| 1.6 | 8.2E-9 |
| 2.2 | 1.6E-7 |
| 2.5 | FAILED |
| | |
| | |

Table 5. (continued) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

| Test 38H (GLT-S) | |
|---|---|
| 350 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 8.4E-9 |
| 0 | 1.0E-8 |
| 0.5 | 6.7E-8 |
| 1.6 | 8.1E-9 |
| 2.9 | 2.0E-8 |
| 3.8 | <9.6E-10 |
| 4.6 | 2.1E-9 |
| 5.3 | 1.2E-9 |
| 5.4 | 3.7E-9 |
| 5.7 | 4.2E-9 |
| 6.1 | 1.0E-8 |
| 6.5 | <9.5E-10 |
| 6.7 | 1.2E-8 |
| 7.0 | <9.1E-10 |
| 7.2 | 2.4E-8 |
| 7.5 | 1.5E-9 |
| 7.9 | 6.0E-9 |
| 8.1 | 1.0E-9 |
| 8.4 | 8.0E-9 |
| 8.8 | 1.0E-9 |
| 9.1 | 4.0E-9 |
| 9.5 | <8.7E-10 |
| 9.7 | <7.6E-10 |
| 10.0 | 1.6E-8 |
| 10.3 | 1.2E-8 |
| 10.8 | 0.6E-9 |
| 11.2 | 9.7E-8 |
| 11.7 | 2.0E-8 |
| 11.9 | FAILED |
| | |
| | |
| | |
| | |

| Test 39H (GLT-S) | |
|---|---|
| 350 ºF, Air fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.7E-9 |
| 0 | 1.1E-8 |
| 0.5 | 6.6E-8 |
| 0.8 | 8.4E-9 |
| 1.6 | 3.1E-9 |
| 2.2 | <9.9E-10 |
| 2.4 | 4.2E-9 |
| 2.8 | 1.0E-8 |
| 3.8 | FAILED |
| | |
| Test 50H | |
| 400 ºF, $CO_2$ fill, 2E05/72 min | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.1E-9 |
| 0 | 9.2E-9 |
| 1 | 5.5E-8 |
| 1.9 | 4.8E-8 |
| 2.9 | 2.9E-9 |
| 3.8 | <9.2E-10 |
| 4.6 | <8.9E-10 |
| 5.3 | 1.1E-9 |
| 5.7 | 1.1E-9 |
| 6.0 | 5.1E-9 |
| 6.3 | 1.0E-8 |
| 6.7 | <9.75E-10 |
| 7.1 | 1.0E-9 |
| 8.7 | 1.6E-8 |
| 8.2 | 3.0E-9 |
| 8.8 | <7.0E-10 |
| 9.1 | 1.6E-7 |
| 9.4 | FAILED |

| Test 60H | |
|---|---|
| 400 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.5E-9 |
| 0 | 1.1E-8 |
| 0.5 | 2.1E-8 |
| 0.8 | 8.6E-9 |
| 1.6 | FAILED |
| | |
| | |
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| | |
| Test 62H | |
| 400 ºF, $CO_2$ fill, No rad. | |
| Time at Temp (months) | Leak Rate (std cc He/sec) |
| Baseline | 9.1E-9 |
| 0 | 1.1E-8 |
| 0.5 | 5.5E-8 |
| 0.8 | 1.2E-8 |
| 1.6 | FAILED |
| | |
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Table 6. Summary of compression set data* from O-ring fixtures

| Fixture ID & History | Time since Opening | Comp. Set – Inner / Outer O-ring | Time since Opening | Comp. Set – Inner / Outer O-ring | Time since Opening | Comp. Set – Inner / Outer O-ring |
|---|---|---|---|---|---|---|
| *GLT O-Ring Fixtures reported previously with high temperature leak test difficulties* | | | | | | |
| 2 (392 days at 300 °F) | | | | | 30 days | 62% / 59% |
| 29 (283 days at 200 °F) | | | | | 30 days | 30% / 18% |
| 38 (473 days at 300 °F) | <30 min. | 85% / 87% | | | | |
| 39 (456 days at 300 °F) | <30 min. | 77% / 81% | | | | |
| 45 (291 days at 300 °F) | | | | | 30 days | 60% / 71% |
| 46 (493 days at 300 °F) | <30 min. | 76% / 75% | | | | |
| 47 (394 days at 300 °F) | 1 hour | 80% / 81% | 5 days | 77% / 73% | 34 days | 68% / 72% |
| 48 (490 days at 300 °F) | <30 min. | 84% / 84% | | | | |
| 50 (265 days at 300 °F) | | | | | 30 days | 42% / 38% |
| 60 (454 days at 300 °F) | <30 min. | 88% / 89% | | | | |
| 62 (282 days at 300 °F) | | | | | 30 days | 50% / 54% |
| *GLT O-Ring Fixtures removed due to overheating* | | | | | | |
| 16 (522 days at 200 °F) Overheated to >430 °F | 4 hours | 72% / 46% | 10 days | 31% / 28% | 230 days | 29% / 23% |
| 23 (490 days at 200 °F) Overheated to >432 °F | 4 hours | 70% / 89% | 11 days | 33% / 37% | 230 days | 29% / 32% |
| 24 (486 days at 200 °F) Overheated to >342 °F | 4 hours | 75% / 58% | 10 days | 23% / 31% | 230 days | 17% / 24% |
| 34 (654 days at 200 °F) Overheated to >721 °F | 4 hours | 123% / 115% | 11 days | 101% / 86% | 230 days | 101% / 87% |
| *GLT O-Ring Fixtures removed after failing room temperature leak test* | | | | | | |
| 14D (45 days at 400 °F) | 21 minutes | 51% / 77% | 9 days | 54% / 74% | 85 days | 45% / 66% |
| 21D (27 days at 400 °F) | 27 minutes | 66% / 77% | 9 days | 58% / 69% | 80 days | 53% / 66% |
| 23D (12 days at 450 °F) | 21 minutes | 65% / 70% | 14 days | 53% / 63% | 90 days | 54% / 59% |
| 31 (1292 days at 300 °F) | 15 minutes | 84% / 78% | 14 days | 80% / 67% | 31 days | 78% / 65% |
| 32 (1352 days at 300 °F) | 14 minutes | 93% / 83% | 14 days | 90% / 73% | 31 days | 89% / 71% |
| *GLT O-Ring* Fixtures removed for other reasons | | | | | | |
| 28 (630 days at 200 °F) | 4 hours | 68% / 62% | 10 days | 31% / 28% | 230 days | 28% / 24% |
| 62-2007 (~6 months at 300 °F) | 4 hours | 66% / 77% | 11 days | 35% / 35% | 230 days | 32% / 31% |
| *GLT-S O-Ring Fixtures removed after failing room temperature leak test* | | | | | | |
| 28H (50 days at 400 °F) | 10 minutes | 84% / 91% | 11 days | 80% / 88% | 26 days | 80% /88% |
| 38H (358 days at 350 °F) | 20 minutes | 92% / 92% | 14 days | 90% /88% | 30 days | 88% /87% |
| 39H (114 days at 350 °F) | 15 minutes | 78% /90% | 11 days | 74% /89% | 26 days | 72% /88% |
| 45H (99 days at 400 °F) | 12 minutes | 93% /93% | 11 days | 91% /92% | 26 days | 91% /91% |
| 50H (281 days at 400 °F) | 14 minutes | 95% /82% | 14 days | 93% /76% | 30 days | 93% /76% |
| 58H (75 days at 400 °F) | 10 minutes | 83% /87% | 11 days | 81% /84% | 26 days | 78% /84% |
| 60H (50 days at 400 °F) | 7 minutes | 84% /93% | 11 days | 80% /90% | 26 days | 79% /89% |
| 62H (50 days at 400 °F) | 7 minutes | 89% /91% | 11 days | 86% /89% | 26 days | 85% /89% |

* Compression set is calculated per ASTM D395, Method B, as follows:

$$\text{comp. set (\%)} = (t_i - t_f) / (t_i - \text{groove depth}) * 100$$

If the initial radial thickness was not recorded, 0.139 inch is assumed.

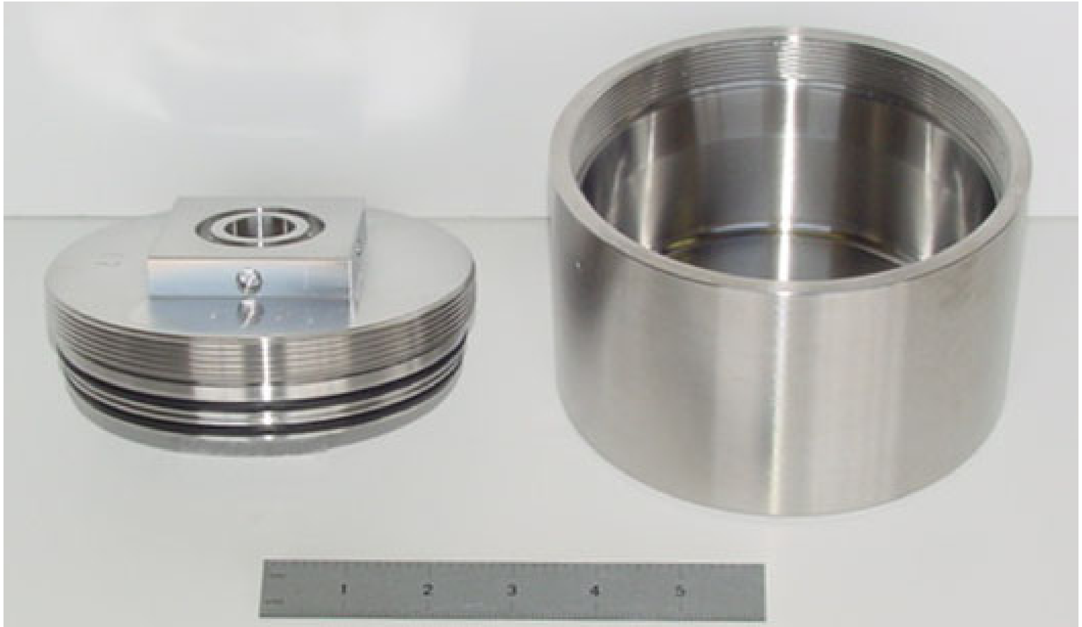



Figure 1. Mock-up PCV test fixture lid and body

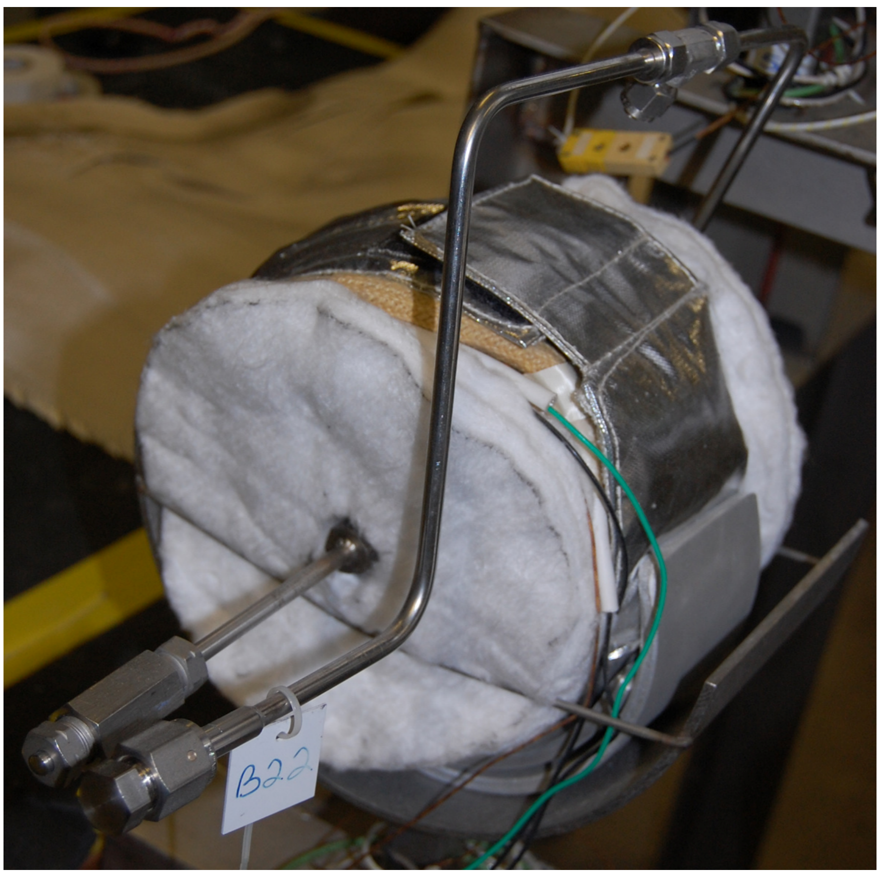



Figure 2. Assembled mock-up PCV

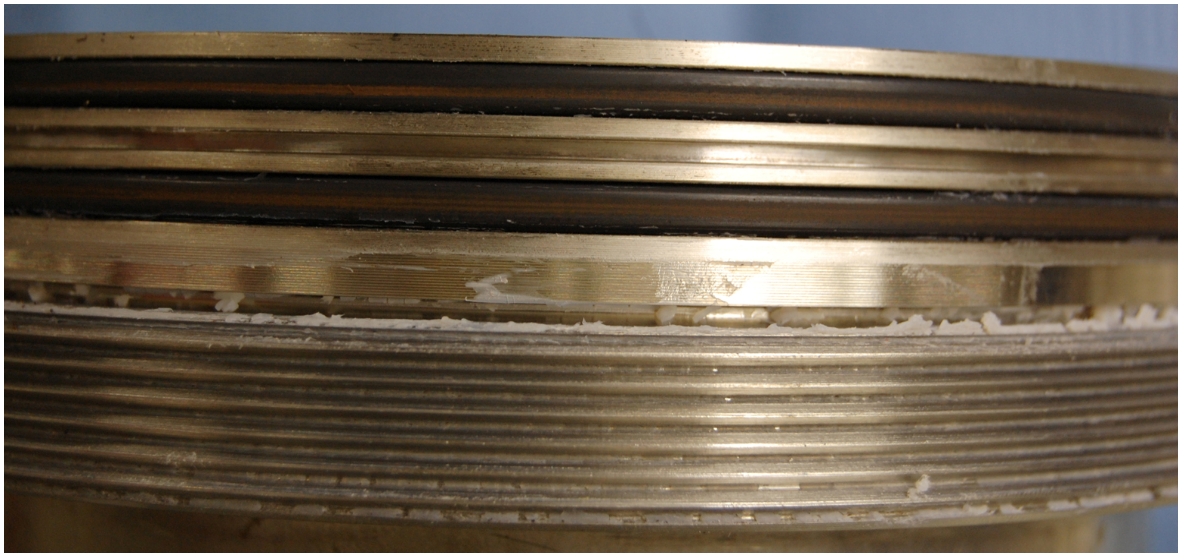
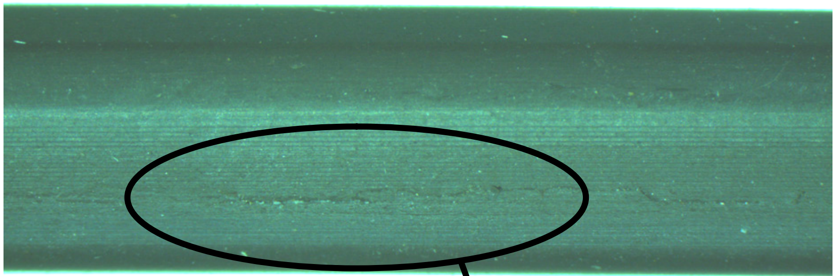
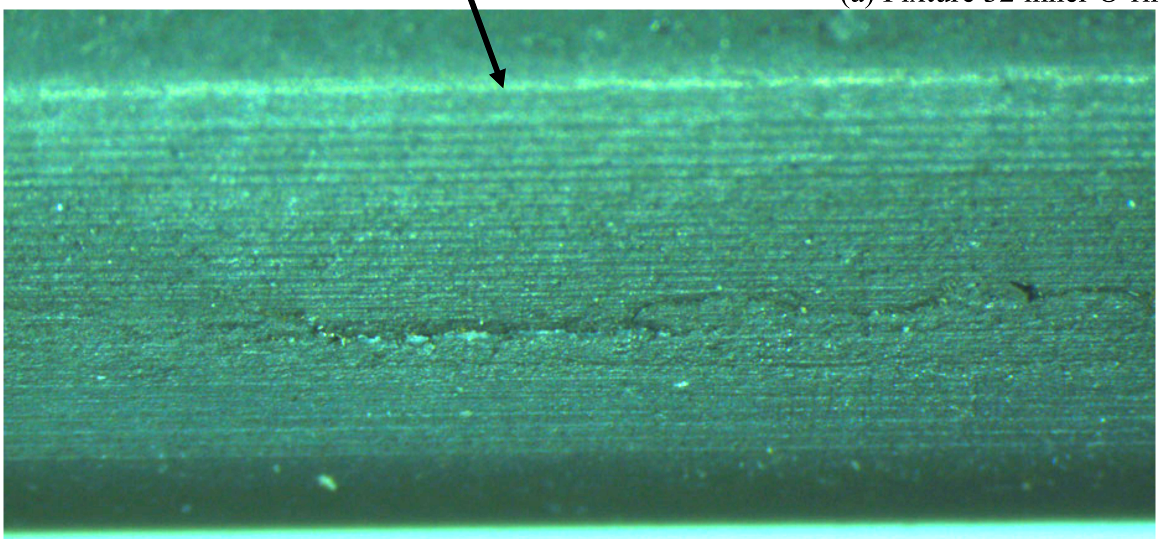


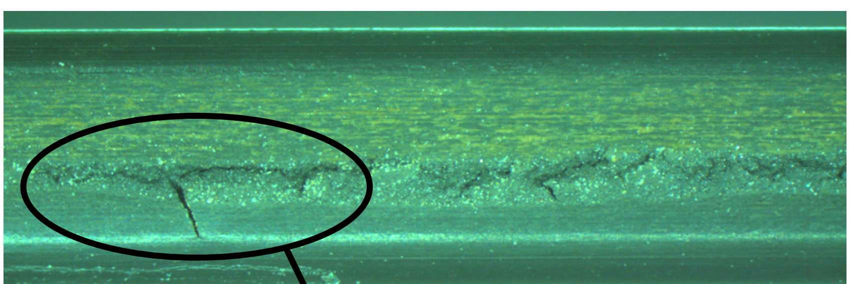
Figure 3. Excess grease on the closure nut from fixture 31. Note also the brown band on each O-ring indicative of oxidized grease (not the same grease used on the closure nut).

Figure 4. Black deposits left on the body of fixture 31 from the O-rings



(a) Fixture 32 inner O-ring





(b) Fixture 32 outer O-ring

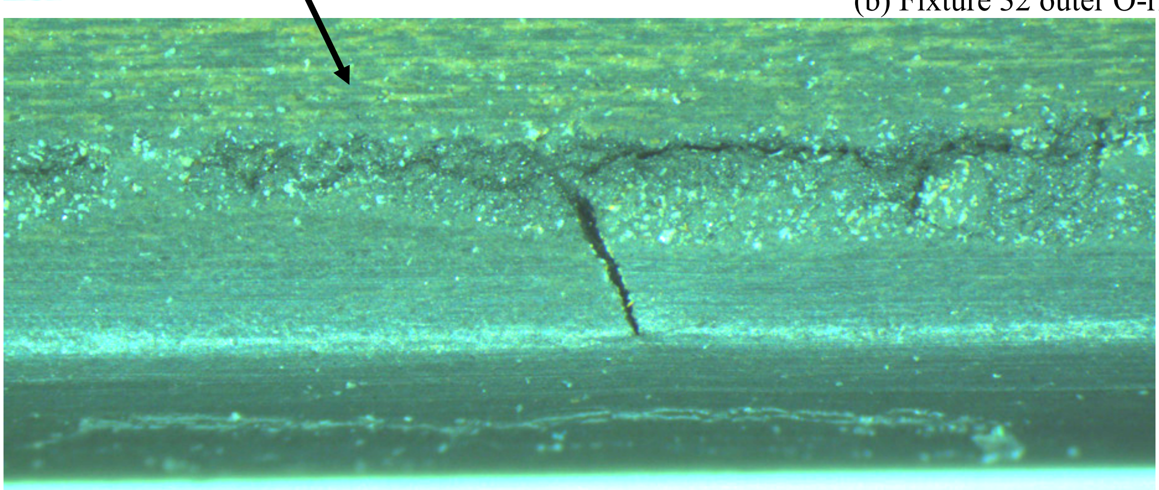


Figure 5. Fixture 32 O-ring ID surfaces showing material loss and/or cracking.

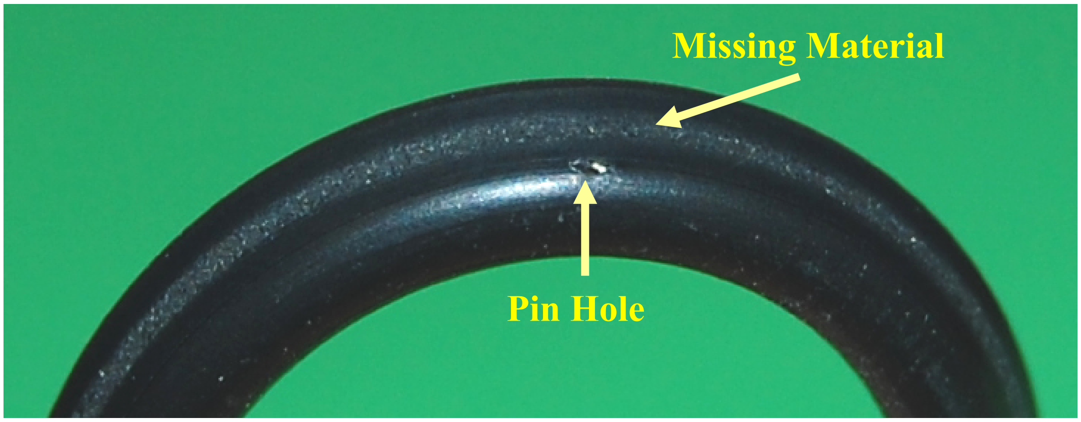



Figure 6. Figure 38H outer O-ring ID surfaces showing material loss and/or cracking and pin hole.

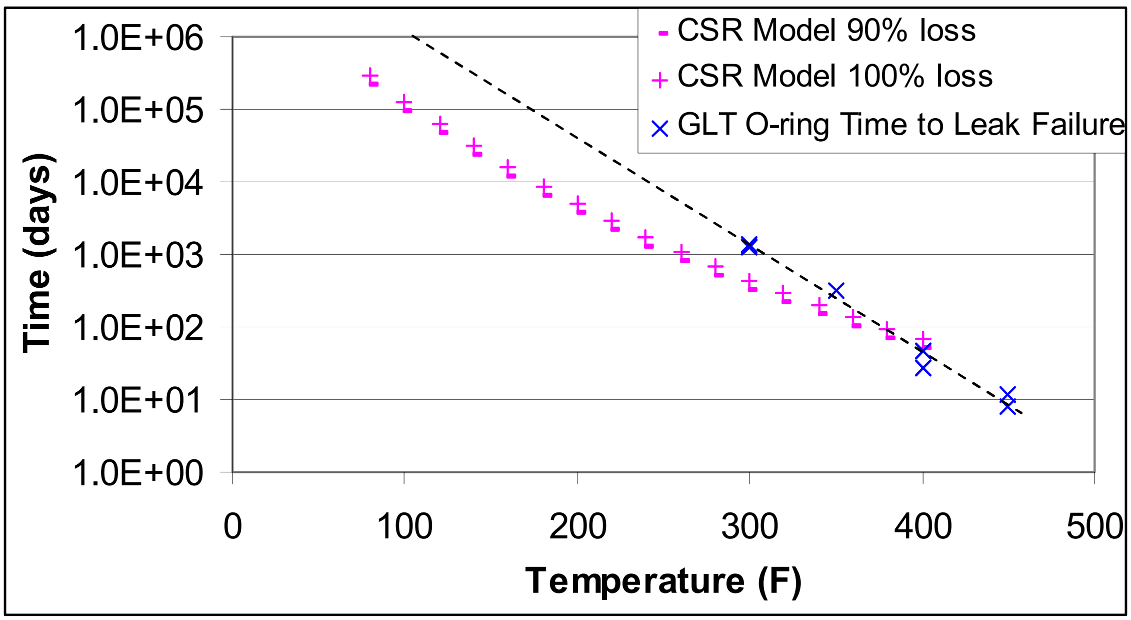



Figure 7 Leakage-based service life data for GLT O-rings (from fixtures with room temperature leak rate failures) compared to life predictions from GLT O-ring compression stress relaxation (CSR) data.

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K. A. Dunn, 773-41A
B. A. Eberhard, 105-K
E. R. Hackney, 705-K
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D. R. LeDuc, 773-41A
J. W. McClard, 705-K
J. W. McEvoy, 730-4B
D. L. Melvin, 705-H
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