

**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 °F. 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 °F.

High temperature aging continues for 36 GLT O-ring fixtures at 200 – 350 °F. Room temperature leak test failures have been experienced in 6 of the GLT O-ring fixtures aging at 300 and 350 °F, 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 still bounding to O-ring temperatures during storage in KAMS.

High temperature aging continues for 6 GLT-S O-ring fixtures at 200 – 300 °F. Room temperature leak test failures have been experienced in all 8 of the GLT-S O-ring fixtures aging at 350 and 400 °F. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 or 300 °F 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 °F). While there are no comparable failure data yet at aging temperatures below 300 °F, 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 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.

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 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 °F, Viton[®] GLT O-rings aged at 350 – 450 °F, and Viton[®] GLT-S O-rings aged at 200 – 400 °F.

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 CO₂, 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 CO₂ 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 °F 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 °F 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 6 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 4 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 °F 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). Five of the GLT O-ring fixtures conditioning at 300 °F 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 44 months. The other fixtures conditioning at 300 °F 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 °F failed to remain leaktight at room temperature after 10.6 months. The second 350 °F fixture remains leaktight at room temperature after 15.4 months at temperature. All three GLT O-ring fixtures conditioning at 400 or 450 °F 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 °F, 250 °F, and 300 °F have remained leaktight at room temperature, with total times at temperature of >19 months. Fixtures conditioned at 350 °F failed to remain leaktight at room temperature after a period of 114 and 358 days. Fixtures conditioned at 400 °F with no radiation exposure failed after 50-99 days. The two fixtures conditioned at 400 °F 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. The basis for such ratings is not well documented by the manufacturer, but thermal ratings are typically based on short-term exposures such as 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 O-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 °F. 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 °C to identify the glass transition temperature. Similar samples from a non-aged O-ring and an O-ring aged at ~265 °F 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 reduces 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 °F (84% avg) and GLT O-rings aged at 400 – 450

°F (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 °F (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 °F). 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.

Conclusions

High temperature aging continues for 36 GLT O-ring fixtures at 200 – 350 °F, and for 6 GLT-S O-ring fixtures at 200 – 300 °F. Room temperature leak test failures have been experienced in 6 of the GLT O-ring fixtures aging at 300 and 350 °F, 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 °F. Room temperature leak test failures have been experienced in all 8 of the GLT-S O-ring fixtures aging at 350 and 400 °F. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 or 300 °F 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 °F), 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 °F, 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	

* Fixtures 26, 49, 52 and 18D have 1 failed O-ring. They remain in test pending failure of the second O-ring.

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 (°F)	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
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 °F, Air fill, No rad.			Test 3 200 °F, CO ₂ fill, No rad.			Test 4 300 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<1.3 E-9		Baseline	<2.9 E-9		Baseline	<1.8 E-9	
6	<9.0 E-10		6	1.0 E-7		6	1.6 E-8	
18	8.0 E-9		15	2.7 E-8		12	2.5 E-8	
30	<1.2 E-8		18	<1.2 E-8		19	<8.0 E-9	
36	<1.2 E-8		31	<1.3 E-8		32	<1.0 E-8	
42	<1.2 E-8		37	<2.0 E-9		38	<8.0 E-9	
48	<2.0 E-8		42	4.0 E-9		42	<1.4 E-8	
			48	2.5 E-8		48	<3.0 E-8	
Test 5 200 °F, CO ₂ fill, 2E5Rad /72 min			Test 6 200 °F, CO ₂ fill, 2E5Rad /72 min			Test 7 300 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<2.3 E-9		Baseline	3.3 E-9		Baseline	4.2 E-8	
6	1.1E-7		6	4.0 E-8		6	8.6 E-8	
11	2.0 E-8		11	5.5 E-8		14	<1.1 E-8	
18	<1.2 E-8		18	<1.2 E-8		17	<1.0 E-8	
31	<1.0 E-8		31	<1.8 E-8		33	<1.2 E-8	
37	<6.0 E-9		37	<2.2 E-8		37	2.4 E-9	
42	<1.0 E-8		42	<1.2 E-8		42	<9.7 E-10	
48	<1.2 E-8		48	<2.8 E-8		44	<2.0 E-8	
						48	<1.3 E-8	
Test 8 300 °F, CO ₂ fill, 2E5Rad /72 min			Test 9 200 °F, Air fill, 2E5Rad /72 min			Test 10 200 °F, CO ₂ fill, 2E5Rad /240 hr		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	2.0 E-8		Baseline	5.6 E-9		Baseline	7.4 E-8	
6	1.1 E-7		6	9.9 E-8		6	1.2 E-7	
12	<1.1 E-8		12	4.2 E-8		13	<1.2 E-8	
18	<1.0 E-8		18	<1.8 E-8		26	5.6 E-9	
37	<1.2 E-8		28	<1.6 E-8		33	<8.0 E-9	
42	4.4 E-9		36	<9.5 E-10		36	2.6 E-9	
47	<1.6 E-8		40	1.0 E-7		42	5.4 E-8	
			42	<1.0E-8				

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

Test 11			Test 12			Test 18		
200 °F, Air fill, 1.4E5 Rad /479 hr			300 °F, Air fill, 2E5Rad /72 min			300 °F, CO ₂ fill, 2E5Rad /246 hr		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<1.5 E-9		Baseline	<1.3 E-8		Baseline	<1.5 E-9	
8	<1.3 E-8		5	2.0E-8		7	<1.1 E-8	
14	<8.8 E-9		9	<1.4 E-8		10	<6.0 E-9	
25	<1.0 E-8		20	<1.4 E-8		26	<1.8 E-8	
31	<4.0 E-9		24	1.1 E-8		29	2.6 E-9	
35	4.0 E-9		30	8.7 E-9		36	6.2 E-8	
42	<1.8 E-8					39	*	

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

Test 20			Test 26			Test 27		
300 °F, Air fill, 1.75E5 Rad /562 hr			300 °F, CO ₂ fill, 2E5Rad /72 min			200 °F, Air fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<1.2 E-8		Baseline	(invalid)		Baseline	1.6 E-9	
6	3.0 E-8		6	1.8 E-8		6	1.5 E-8	
13	<4.8 E-8		12	<1.0 E-8		12	<1.0 E-8	
27	<1.8 E-8		29	<2.0 E-8		19	<3.2 E-8	
33	<6.0 E-9		36	<1.0 E-8		24	<1.2 E-8	
42	<7.2 E-10		41	<2.0 E-8		30	<1.4 E-8	
45	<2.0 E-8		42	>9.0 E-5 *				

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

Test 30			Test 31			Test 32		
300 °F, CO ₂ fill, 2E5Rad /300 hr			300 °F, Air fill, 2E5Rad /72 min			300 °F, Air fill, 2E5Rad /300 hr		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	5.6 E-8		Baseline	(invalid)		Baseline	<1.3 E-9	
6	1.5 E-8		6	1.5 E-8		6	5.9 E-8	
11	<1.8 E-8		11	<1.2 E-8		11	<8.0 E-9	
24	<2.0 E-8		24	<1.6 E-8		25	<3.0 E-8	
32	<3.0 E-8		35	2.6 E-8		32	<1.8 E-8	
36	5.3 E-9		42	<2.6 E-8		36	2.6 E-8	
42	2.1 E-9		45	8.2 E-7 *		42	<1.4 E-8	
						44	2.9 E-6 **	

* Test 31 – both O-rings failed at 45 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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, Air fill, 2E5Rad /72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
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			Test 19D		
350 °F, Air fill, 2E5Rad /72 min			350 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<8.0 E-9		Baseline	2.8 E-8	
0.3	1.6 E-8		0.3	<7.9 E-9	
0.9	3.2 E-8		0.9	2.3 E-8	
1.5	<8.1 E-9		1.5	<8.6 E-9	
2.2	<1.9 E-9		2.2	<1.9 E-9	
3.0	5.0 E-8		3.0	2.2 E-8	
3.5	6.1 E-9		3.5	<2.8 E-9	
4.2	9.9 E-10		4.2	1.4 E-9	
4.7	3.6 E-9		4.7	2.0 E-8	
5.4	1.9 E-8		5.3	7.6 E-9	
5.9	1.3 E-9		5.9	1.5 E-9	
6.4	2.0 E-8		6.4	4.8 E-9	
7.1	3.3 E-9		7.1	<8.0 E-9	
7.6	<6.0 E-9		7.6	<1.0 E-8	
8.0	<6.0 E-9		8.0	<1.0 E-8	
8.5	<9.8 E-10		8.5	<1.0 E-9	
8.9	<1.8 E-8		9.0	<2.2 E-8	
9.5	<8.8 E-10		9.6	<8.8 E-10	
10.0	<7.4 E-10		10.1	<7.0 E-10	
10.6	6.0 E-5 **		10.8	3.4 E-8	
11.0	3.8 E-8		11.5	<1.2 E-8	
11.6	<4.6 E-8		14.8	5.9 E-9	
12.3	<2.8 E-8		15.4	1.3 E-8	
12.6	<5.8 E-8		16.0	1.5E-8	
13.2	8.9 E-9				
13.9	9.4 E-8				
14.5	1.8E-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 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<8.5E-09	
0.5	<1.1E-08	
0.9	2.3E-08	
1.5	<9.5E-09	
1.9	<1.1E-09	
2.1	2.3E-09	
2.8	<1.1E-09	
3.4	1.5E-09	
3.9	4.4E-09	
4.4	<8.0E-09	
5.2	2.8E-09	
5.5	<8.0E-09	
6.5	<1.8E-08	
6.9	<8.9E-10	
7.7	<7.2E-10	
8.3	<1.8E-08	
9.0	<1.4E-08	
13.8	<1.0E-08	

Test 15H (GLT-S)		
200 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.8E-09	
0.5	<1.0E-08	
0.9	3.1E-08	
1.5	3.6E-08	
1.8	<2.7E-09	
2.2	3.2E-8	
2.7	1.1E-09	
3.3	<1.2E-09	
3.9	1.1E-09	
4.4	2.9E-09	
5.0	<4.0E-09	
5.7	3.5E-09	
6.1	<4.0E-09	
6.6	<9.1E-10	
7.1	2.4E-08	
7.5	<1.0E-09	
8.2	<7.3E-10	
8.8	<1.8E-08	
9.5	<1.4E-08	
14.3	1.5E-08	

Test 16H (GLT-S)		
250 °F, Air fill, No rad..		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.1E-09	
0.5	<1.1E-08	
0.9	4.4E-07	
1.2	<8.1E-09	
1.6	<9.0E-10	
2.0	1.8E-09	
2.6	<1.2E-09	
3.2	1.5E-09	
3.7	6.6E-09	
4.2	<1.2E-08	
4.9	<8.0E-09	
5.2	<8.0E-09	
5.7	<8.8E-10	
6.2	<1.6E-08	
6.6	<8.6E-10	
7.4	<7.2E-10	
8.0	<1.8E-08	
8.7	1.5E-08	
9.9	<1.6E-08	
11.2	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)			Test 29H (GLT-S)			Test 34H (GLT-S)		
250 °F, Air fill, No rad.			300 °F, Air fill, No rad.			300 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.1E-09		Baseline	<8.2E-09		Baseline	<9.5E-09	
0.4	<1.0E-08		0.5	1.6E-08		0.8	<1.0E-08	
0.9	3.8E-07		1.1	<8.1E-09		1.2	<2.0E-09	
1.2	9.0E-09		1.7	6.2E-09		1.8	<8.0E-09	
1.7	<2.9E-09		2.0	<1.0E-09		2.4	<3.2E-09	
2.1	2.0E-09		2.5	1.5E-09		2.7	<1.0E-09	
2.6	2.6E-09		3.2	<1.1E-09		3.2	2.2E-09	
3.1	<1.0E-09		3.8	2.2E-08		3.8	<1.1E-09	
3.7	8.8E-10		4.2	4.2E-09		4.5	8.8E-10	
4.2	3.7E-09		4.9	9.9E-09		4.9	2.4E-09	
4.8	<8.0E-09		5.5	<9.6E-10		5.6	<1.0E-08	
5.4	<9.6E-10		5.9	<8.0E-09		6.2	<9.5E-09	
5.8	<8.0E-09		6.2	<9.1E-10		6.5	<1.2E-08	
6.4	<9.1E-10		6.4	<2.4E-08		7.1	<9.1E-10	
6.8	<2.8E-08		6.9	<1.1E-09		7.6	<2.4E-08	
7.3	<1.1E-09		7.5	<6.7E-10		8.1	<1.0E-09	
8.0	<7.3E-10		8.2	<1.8E-08		8.7	<6.8E-10	
8.6	<2.0E-08		8.8	1.2E-08		9.3	<1.6E-08	
9.3	<2.6E-08		9.8	<2.0E-08		10.0	<1.6E-08	
10.6	<1.8E-08		11.0	2.9E-08		11.0	<1.6E-08	
12.0	<2.0E-08		12.3	<1.0E-08		11.9	<3.0E-08	
13.3	3.9E-08					13.1	<1.2E-09	

Test 28H (GLT-S)			Test 45H			Test 58H		
400 °F, CO ₂ fill, 2E05 in 72 min			400 °F, Air fill, No rad.			400 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc	Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.1E-9		Baseline	<9.3E-9		Baseline	1.5E-8	
0.5	1.9E-9		0.5	<1.9E-9		0.5	5.1E-8	
1.1	4.9E-8		1.1	**		1.1	<7.8E-9	
1.7	FAILED*		1.6	2.1E-9		1.6	8.2E-9	
			2.1	<1.1E-9		2.2	1.6E-7	
			3.3	FAILED**		2.5	FAILED***	

*Test 28H – both O-rings failed at 1.6 months

**Test 45H – outer O-ring failed at 0.8 months. Subsequent results on inner O-ring only. Inner O-ring failed at 3.3 months.

***Test 58H – both O-rings failed at 2.5 months

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

Test 38H (GLT-S)		
350 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<8.4E-9	
0.5	6.7E-8	
1.1	<8.1E-9	
1.7	2.0E-8	
2.0	<9.6E-10	
2.5	2.1E-9	
3.2	<1.2E-9	
3.8	3.7E-9	
4.2	4.2E-9	
4.9	<6.0E-9	
5.5	<1.0E-9	
5.9	<8.0E-9	
6.4	<1.0E-9	
6.9	<4.0E-9	
7.4	<8.7E-10	
8.0	<7.6E-10	
8.7	<1.6E-8	
9.3	<1.2E-8	
10.0	9.7E-8	
10.6	<6.0E-10	
11.2	<1.6E-8	
11.8	FAILED*	

*Test 38H – both O-rings
failed at 11.8 months

Test 39H (GLT-S)		
350 °F, Air fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.7E-9	
0.5	6.6E-8	
1.1	<8.4E-9	
1.7	3.1E-9	
2.0	9.9E-10	
2.5	4.2E-9	
3.1	1.0E-8	
3.8	FAILED*	

*Test 39H – both O-rings failed at 3.8 months

**Test 60H – both O-rings failed at 1.6 months

Test 60H		
400 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.5E-9	
0.5	2.1E-8	
1.1	<8.6E-9	
1.6	FAILED**	

Test 50H		
400 °F, CO ₂ fill, 2E05/72 min		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.1E-9	
0.5	5.5E-8	
1.1	4.8E-8	
1.6	<2.9E-9	
2.0	<9.2E-10	
2.4	<8.9E-10	
3.1	<1.1E-9	
3.7	1.1E-9	
4.2	5.1E-9	
4.8	<1.0E-8	
5.4	<9.7E-10	
5.8	<1.0E-9	
6.3	<9.3E-10	
6.8	<1.6E-8	
7.3	3.0E-9	
7.9	<7.0E-10	
8.6	1.6E-7	
9.2	FAILED*	

*Test50H – both O-rings failed at 9.2 months

Test 62H		
400 °F, CO ₂ fill, No rad.		
Time at Temp (months)	Leak (std He/sec)	Rate cc
Baseline	<9.1E-9	
0.5	5.5E-8	
1.1	<1.2E-8	
1.6	FAILED*	

*Test 62H – both O-rings
failed at 1.6 months

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
<i>GLT O-Ring Fixtures reported previously with high temperature leak test difficulties</i>						
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%
<i>GLT O-Ring Fixtures removed due to overheating</i>						
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%
<i>GLT O-Ring Fixtures removed after failing room temperature leak test</i>						
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%
<i>GLT O-Ring Fixtures removed for other reasons</i>						
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%
<i>GLT-S O-Ring Fixtures removed after failing room temperature leak test</i>						
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.

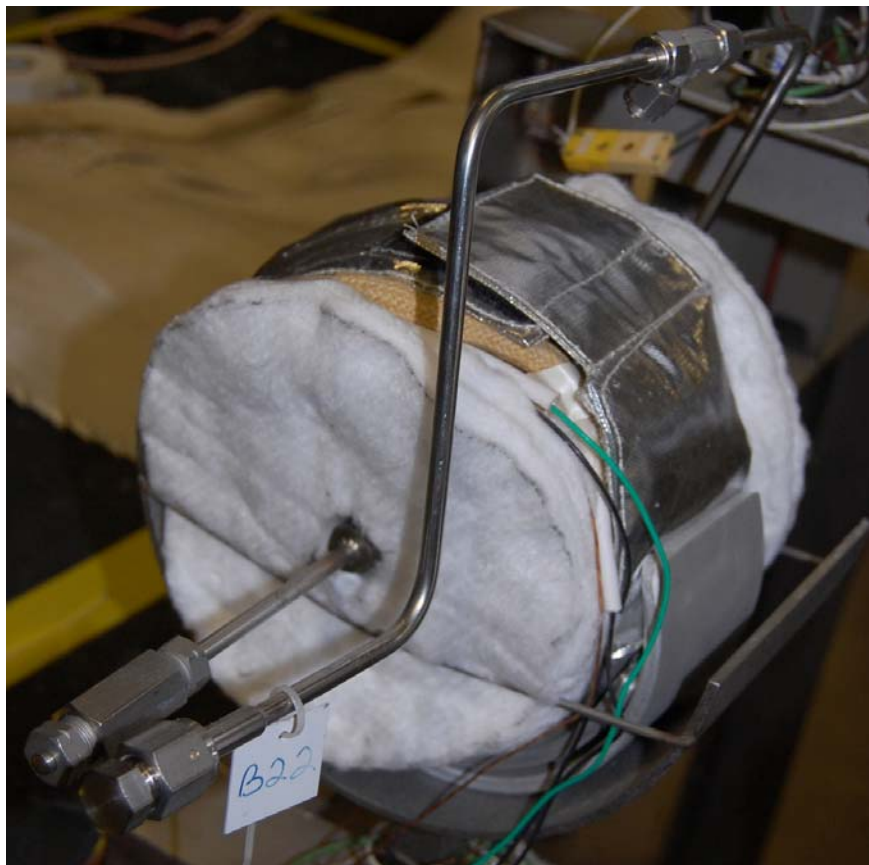


Figure 2. Assembled mock-up PCV.

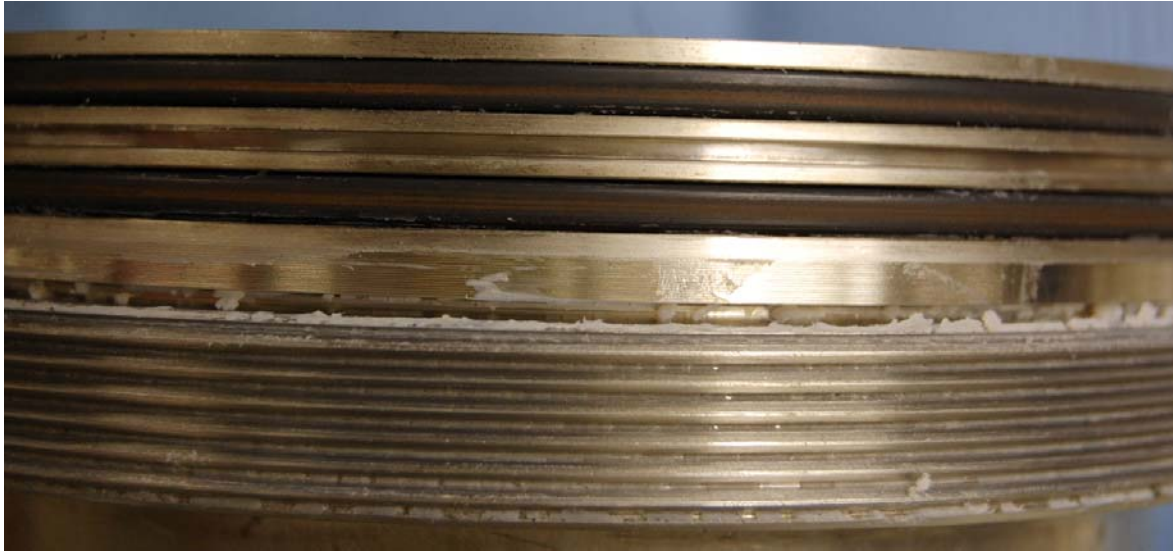


Figure 3. Excess grease on the closure nut from fixture 31. Note also the brown band on each O-ring indicative of oxidized grease (not the same grease used on the closure nut).



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

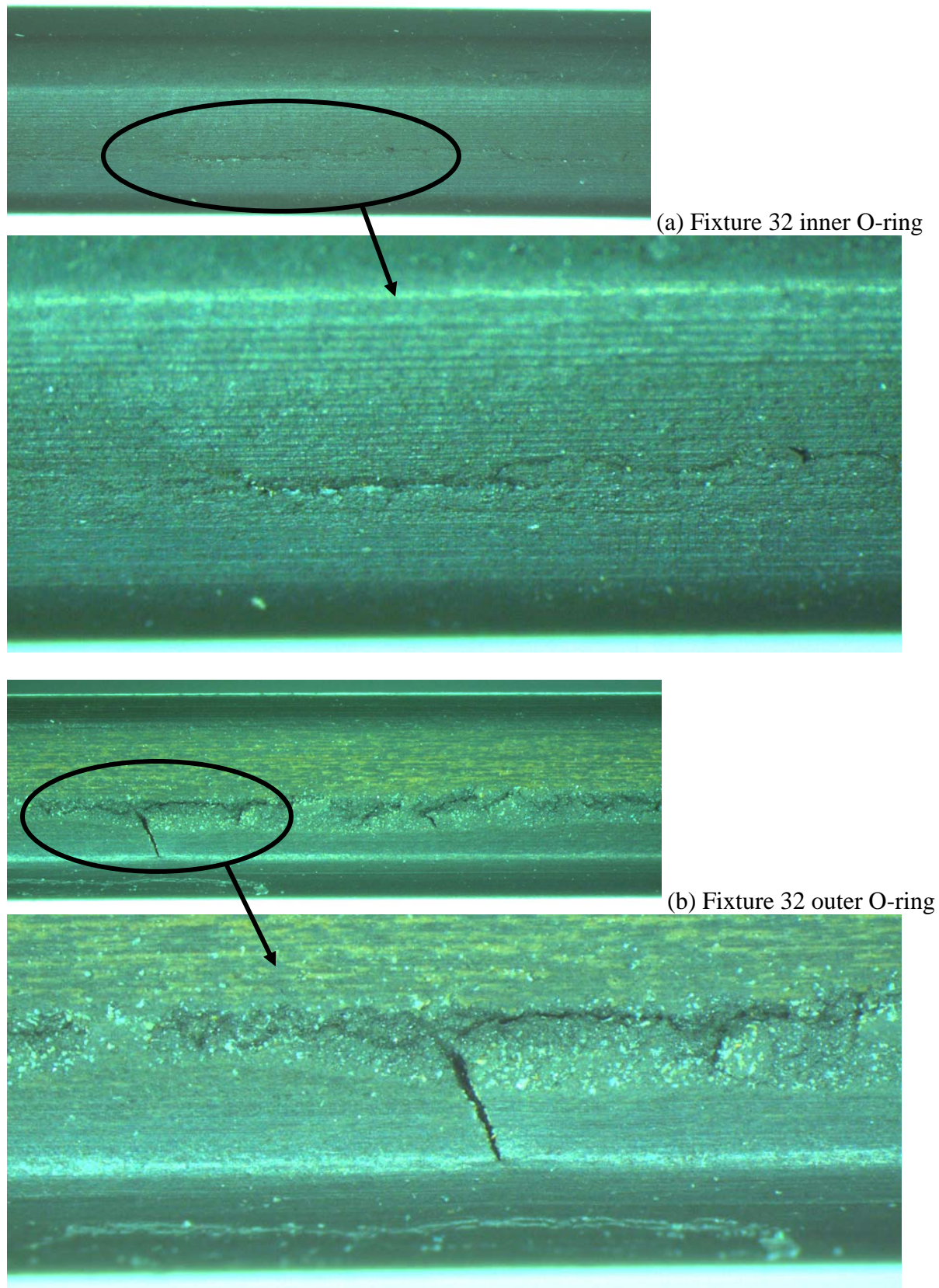


Figure 5. Fixture 32 O-ring ID surfaces showing material loss and/or cracking.

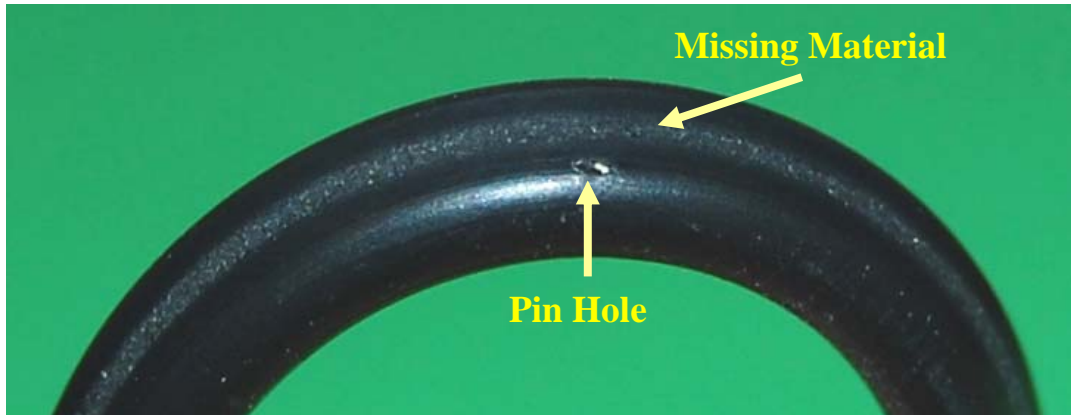


Figure 6. Figure 38H outer O-ring ID surfaces showing material loss and/or cracking and pin hole.

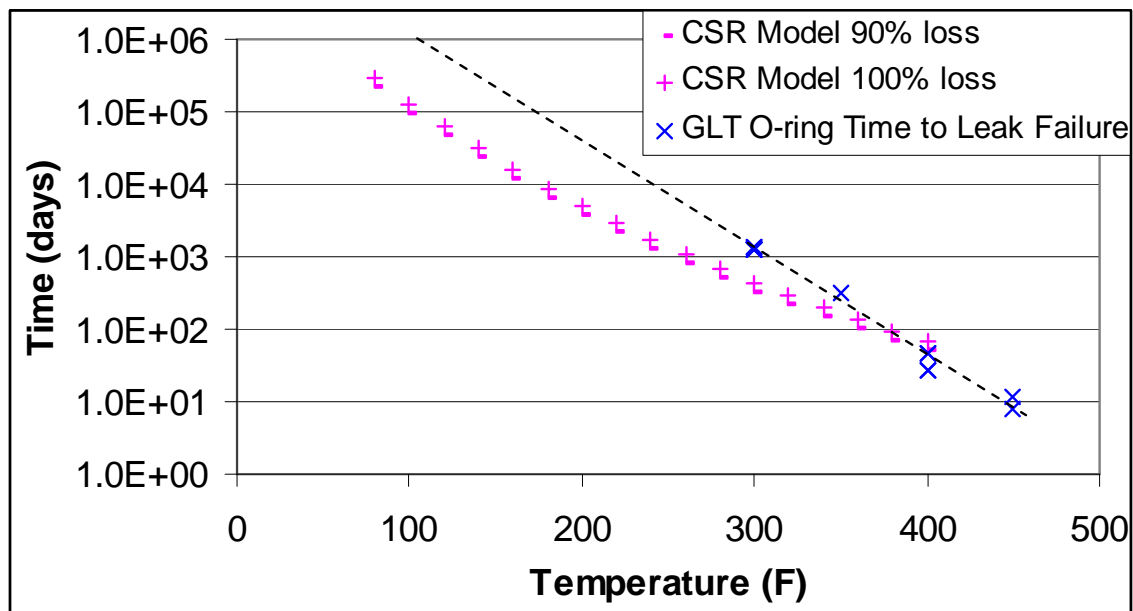


Figure 7 Leakage-based service life data for GLT O-rings (from fixtures with room temperature leak rate failures) compared to life predictions from GLT O-ring compression stress relaxation (CSR) data.

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