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

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Seventh 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 since 2004 at the Savannah River National Laboratory. Seventy tests using 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 periodically to determine if they meet the criterion of leak-tightness 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 23 GLT O-ring fixtures at 200 – 270 ºF. Room temperature leak test failures have been experienced in all of the GLT O-ring fixtures aging at 350 ºF and higher temperatures, and in 8 fixtures aging at 300 ºF. The remaining GLT O-ring fixtures aging at 300 ºF have been retired from testing following more than 5 years at temperature without failure. No failures have yet been observed in GLT O-ring fixtures aging at 200 ºF for 54-72 months, which is still bounding to O-ring temperatures during storage in K-Area Complex (KAC). Based on expectations that the fixtures aging at 200 ºF will remain leak-tight for a significant period yet to come, 2 additional fixtures began aging in 2011 at an intermediate temperature of 270 ºF, with hopes that they may reach a failure condition before the 200 ºF fixtures.

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 - 300 ºF for 30 - 36 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 – 96%. This is greater than seen to date for any packages inspected during KAC field surveillance (24% average).

For GLT O-rings, separate service life estimates have been made based on the O-ring fixture leak test data and based on compression stress relaxation (CSR) data. These two predictive models show reasonable agreement at higher temperatures (350 – 400 ºF). However, at 300 ºF, the room temperature leak test failures to date experienced longer aging times than predicted by the CSR-based model. This suggests that extrapolations of the CSR model predictions to temperatures below 300 ºF will provide a conservative prediction of service life relative to the leak rate criterion. Leak test 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 PCV O-ring 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 KAC 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 KAC. O-ring performance in these tests is defined by leak-tightness, per ANSI standard N14.5-97 at room temperature.

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 ºF, Viton® GLT O-rings aged at 270 – 450 ºF, and Viton® GLT-S O-rings aged at 200 – 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 KAC 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 (internal PCV atmosphere was subsequently dropped as a test variable). 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 or separately. With this arrangement, data are obtained on both O-rings installed in each fixture. Although only the outer O-ring is credited for containment, testing both provides twice the information under nearly identical conditions.

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. Several fixtures conditioning at 300 ºF have experienced room temperature O-ring leakage (i.e. failed), beginning in April 2010. 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 were leak tested on a nominal 3 week frequency.

An additional two fixtures with Viton® GLT O-rings were added to the second test matrix in April 2011, and began aging at 270 °F. With leak test failures experienced at aging temperatures of 300 °F and above, and no failures projected to occur at 200 °F for many years yet, it was anticipated that these two intermediate temperature fixtures might experience leak failures sooner that the 200 °F fixtures. This would provide additional confirmation of the extrapolation model for leak test data at an earlier date than the 200 °F fixtures.

All of the second matrix 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 (approximating a 10-year service dose at a bounding rate of 2 rad/hr).

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.

Fixture Leak Testing

The O-ring fixtures are leak-tested after initial setup, after irradiation, and periodically thereafter to the same leak-tight criterion as the 9975 PCV and SCV. The outer O-rings of the 9975 PCV and SCV are credited with being leak-tight while in transport and are credited with maintaining containment while in storage in the KAC [5, 6].

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

Leak testing is conducted using a Varian 959 helium mass spectrometer leak detector. 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 [7], 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 [8].

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 8), subsequent testing is conducted without the extended duration to demonstrate permeation, since no actions are performed that might disrupt the flow path during aging and leak testing. All fixtures in test since December 2008 have demonstrated permeation.

Results

Mock-up PCVs have been assembled and aged in the conductance of 70 tests on GLT O-rings, and 14 tests on GLT-S O-rings. This report summarizes results for these fixtures through July 1, 2012.

A total of 23 GLT O-ring fixtures and 6 GLT-S O-ring fixtures remain in test. All of the GLT O-ring fixtures conditioning at 200 ºF have remained leak-tight at room temperature, with total times at temperature ranging from 54 to 72 months (at the time of their last leak test). Eight of the GLT O-ring fixtures conditioning at 300 ºF have failed to remain leak-tight (in one or both O-rings) at room temperature. Failure of these was identified at exposure times ranging from 34 to 68 months. The other GLT fixtures conditioning at 300 ºF were retired from test in July 2012 with exposure times ranging from 61 to 74 months. Each was given a final leak test, and permeation of helium through the O-rings was re-verified. The GLT O-rings in fixtures aging at 350 F and higher failed previously, as noted in prior status reports. The times to failure for each GLT O-ring fixture are summarized in Table 3. Detailed leak rate histories can be found in Table 4 for fixtures in test since the last status report [9].

All of the GLT-S O-ring fixtures conditioning at 200 ºF, 250 ºF, and 300 ºF have remained leak-tight at room temperature, with total times at temperature of 30 to 36 months. GLT-S fixtures conditioned at 350 ºF and at 400 ºF failed as noted in previous status reports. The times to failure for each GLT-S O-ring fixture are summarized in Table 3. Detailed leak rate histories can be found in Table 5 for fixtures in test since the last status report [9].

Fixture 8 was aged at 300 °F for 2082 days, and was disassembled in February 2012 following failure of both GLT O-rings. The following observations were made during disassembly:

- The inner O-ring was tightly adhered to the plug in one area. The outer O-ring was tightly adhered to the plug over the entire circumference.
- A black stain and/or some adhering O-ring material was observed on both the fixture body and plug where the O-rings contacted them. See Figure 3
- Both O-rings experienced material pullout from the surface facing the bottom of the O-ring groove. Some cracking was also observed on this surface of the outer O-ring. See Figure 4.

Both O-rings removed from fixture 8 received dimensional and hardness measurements. Average hardness readings for each O-ring were 78 to 79 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 (~2 points) from baseline measurements for each of these O-rings. This difference is not considered significant. Aside from the cracking and tearout observed on the O-rings, which likely resulted from removal, there was no obvious indication of defect or cause of failure.

The dimensional measurements were used to calculate compression set. The measurements were taken within 30 minutes of removal, and again after ~14 and 30 days. These measurement intervals are generally consistent with that used previously on other fixtures. Compression set results are summarized in Table 5.

Discussion

In July 2012, there were 9 remaining fixtures with GLT O-rings conditioning at 300 ºF. These were given a final leak test, and retired from test. During the final leak test, one of these fixtures (fixture 33 inner O-ring only, the outer O-ring had previously failed) failed the leak test, and the remaining 8 were still leak-tight. Aging times for these fixtures ranged from 61 to 74 months. It was reasoned that the continued aging of these fixtures would no longer contribute to extending the O-ring service life since O-rings aged at 300 ºF had previously failed after exposure times as short as 34 months. It is anticipated that some of these eight fixtures will be opened for a final examination, while the rest will be maintained as-is for potential future test initiatives. Given the behavior observed in fixture 8 and other fixtures previously disassembled, it is considered likely that adhesion of the O-rings to the vessel has contributed to the continued leak-tightness of the last eight GLT fixtures aging at 300 ºF.

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. All fixtures in test since December 2008 have shown permeation.

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. leak-tight for reasons other than seal performance). An open flow path has now been demonstrated for all fixtures. In general, they will not be tested to permeation in future leak tests, although the 300 ºF GLT fixtures that were retired from testing in July 2012 received a final leak test and permeation re-verification. 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. For the eight 300 ºF fixtures which have been retired from test, the final leak test was extended to again demonstrate a permeation signal.

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 96%, with an average value of 85%, for measurements within the first 30 minutes. The average compression set was slightly higher for the GLT-S O-rings (89%) than for the GLT O-rings (82%). There is a difference between the compression set for GLT O-rings aged at 300 – 350 ºF (87% 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 compression set measured on SCV and PCV O-rings during field surveillance to date ranges from -5 to 50% (with an average of 24%), following storage periods in KAC of up to 7 years. Some of this variation results from using the assumed initial O-ring thickness of 0.139 inch, since no data exist on the baseline conditions of these O-rings. The average compression set for these O-rings removed from service 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.

The compression set data are summarized in Figures 5 and 6. Figure 5 presents results from measurements taken immediately after opening the vessel (typically within 30 minutes), and includes 2 sets of data from test fixtures with GLT O-rings aged at 300-450 ºF. In the first data set, fixtures that failed to remain leak-tight have compression set values of 70 – 93% (300 ºF fixtures) or 51 – 96% (350 – 450 ºF fixtures). The second set consists of 300 ºF fixtures that were removed from test due to difficulties with high temperature leak testing, but that remained leak-tight at room temperature. These O-rings have compression set values of 75 – 89%, even though they were at temperature for a much shorter time than the first set. This indicates that a high compression set value is not necessarily an indication of imminent leak failure. In contrast, Figure 5 also shows the compression set data from field surveillance of SCV and PCV O-rings. The field surveillance data show a wide range of scatter due in part to the assumption of an initial nominal O-ring thickness. Nevertheless, the range of compression set data from field surveillance remains consistently at 50% or less, with time in service ranging up to ~7 years. Only 1% of the field surveillance compression set values are greater than 40%, so the typical compression set for O-rings in service to date is significantly less than that observed in O-rings which have failed to remain leak-tight. It is currently unknown whether the compression set for O-rings in service will gradually increase to a higher value prior to their failure, but the available evidence strongly indicates the O-rings in service are not yet close to reaching an end-of-life condition.

The compression set data in Figure 6 include results from all fixtures that have failed to remain leak-tight, and show results based on re-measurements taken 26 – 90 days after opening the vessels. By this time, the O-rings have recovered about as much of their original dimensions as is likely. Results for the GLT O-rings appear to fall into separate groupings based on aging temperature, with different behavior at 300 ºF than that at 350 ºF or 400 – 450 ºF. The compression set data for GLT-S O-rings at 350 and 400 ºF are intermediate to the GLT data for these same temperatures.

A separate task [2, 10] is underway to correlate the compression stress relaxation (CSR) of O-rings with aging temperature. CSR testing tracks material behavior independent of the factors involved in leak testing, and it is hoped that the time for the compressive force to relax to a specific value (such as 90% 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 11. With several room temperature leak failures of aged O-ring fixtures, an initial comparison is now possible to demonstrate whether CSR data correlate 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 average O-ring temperature in storage is significantly lower than the temperature range for either the CSR or fixture data. Insufficient data currently exist to draw a similar comparison for GLT-S O-rings.

Conclusions

High temperature aging continues for 23 GLT O-ring fixtures at 200 – 270 ºF. Room temperature leak test failures have been experienced in 8 of the GLT O-ring fixtures aging at 300 ºF, and in all 5 of the GLT O-ring fixtures aging at higher temperatures. The remaining 8 GLT O-ring fixtures aging at 300 F were retired from testing following at least 61 months at temperature. No failures have yet been observed in GLT O-ring fixtures aging at 200 ºF for 54-72 months, which is more representative of (but still bounding to) O-ring temperatures during storage in KAC. The maximum O-ring temperature possible to date in the KAC is ~ 176 °F, based on a transient peak ambient temperature of 106 °F and the maximum payload (19W). Average ambient temperatures and reduced payloads are less challenging to the seals in storage.

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 - 300 ºF for 30 – 36 months.

For one GLT O-ring fixture that failed the room temperature leak test and was disassembled within the past year, the O-rings displayed a compression set of 90 – 92%. These values are higher than seen to date for packages inspected during KAMS field surveillance (24% 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 KAC 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. A CSR aging model for GLT-S O-rings is in progress and will be reported separately.

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

References

[1] WSRC-TR-2003-00325, Rev. 4, "Task Technical and Quality Assurance Plan for Characterization and Surveillance of Model 9975 Package O-Rings and Celotex® Materials (U)", January 2009.

[2] SRNS-TR-2008-00054, Rev. 0, "Task Technical and Quality Assurance Plan for Accelerated Aging of Viton® GLT-S O-rings for Model 9975 Shipping Packages in KAMS (U)", January 2009.

[3] WSRC-TR-2001-0286, Rev. 4, "SRS Surveillance Program for Storage of Pu Material in KAMS", July 2008.

[4] SRNL-TR-2009-00186, "Fourth Interim Status Report: Model 9975 PCV O-Ring Fixture Long-Term Leak Performance", W. L. Daugherty and T. M. Stefek, June 2009

[5] WSRC-SA-2002-00008, Rev. 0, "Safety Analysis Report for Packaging – Model 9975", December 2003.

[6] L9.4-10500, Rev. 2, "Annual Maintenance and Leak Testing for the 9975 Shipping Package," Instrumentation and Equipment Systems Section, SRNL, August 29, 2007.

[7] ANSI Standard N14.5-97, "American National Standard for Radioactive Materials - Leakage Tests on Packages for Shipment", American National Standards Institute, New York, NY, February 1998.

[8] SRNL-L1400-2008-00038, "Characterization of Leak Test System for O-Ring Studies", D. J. Trapp, December 18, 2008.

[9] SRNL-TR-2011-00217, "Sixth Interim Status Report: Model 9975 PCV O-Ring Fixture Long-Term Leak Performance", W. L. Daugherty, August 2011.

[10] WSRC-TR-2007-00276, Rev. 0, "Task Technical and Quality Assurance Plan for Model 9975 O-ring Life Prediction (U)", January 2008.

[11] SRNS-TR-2009-00259, "Lifetime Prediction for Model 9975 O-Rings in KAMS", E. N. Hoffman and T. E. Skidmore, November 2009.

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 KAC 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].
	270, 350, 400, 450 °F (132, 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 (the initial period to be validated).
	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.
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.

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	Retired July 2012	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		8, 12, 26, 31	7, 51, 52	17, 22, 25, 39, 45, 46, 47, 58, 59, 60
300	~2E5 Low	Normal		32	18, 30*	21, 38
300	No	Normal		49, 33	4, 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						
270	No	Normal	14W			
270	No	Normal	21W			
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 High	Normal		28H, 50H		

* Fixture 30 has 1 failed O-ring (inner).

Table 3. Summary of GLT and GLT-S O-ring leak failures

		Days at temperature to failure *	
Fixture	**Temp (ºF)**	**Inner**	**Outer**
GLT O-ring Fixtures			
8	300	2009 - 2082	2009 - 2082
12	300	957 - 1020	957 - 1020
26	300	1273 - 1366	1261 - 1273
30	300	1279 - 1392	N A
31	300	1280 - 1291	1280 - 1291
32	300	1271 - 1352	1271 - 1352
33	300	1360 - 1466	1924 - 1979
49	300	1101 - 1276	1323 - 1360
18D	350	481 - 497	304 - 324
19D	350	573 - 594	560 - 571
14D	400	29 - 45	29 - 45
21D	400	8 - 28	8 - 28
23D	450	10 - 12	0 - 8
GLT-S O-ring Fixtures			
38H	350	338 - 358	338 - 358
39H	350	95 - 114	95 - 114
45H	400	65 - 99	14 - 33
58H	400	62 - 75	62 - 75
60H	400	33 - 50	33 - 50
62H	400	34 - 50	34 - 50
28H	400	33 - 50	33 - 50
50H	400	260 - 281	260 - 281

* The first time at temperature is the last successful leak test. The second time at temperature is the failed leak test. Failure occurred at some point between these two times.

Table 4. Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 1, 200 ºF	
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
54	<1.0 E-8
60	<3.0 E-8
66	<3.0 E-8
72	<2.0 E-9

Test 3, 200 ºF	
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
54	<1.3 E-8
59	<2.8 E-8
65	<1.8 E-8
72	<1.6 E-8

Test 5, 200 ºF	
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
53	<1.0 E-8
59	<2.4 E-8
66	<2.6 E-8
71	1.0 E-7

Test 6, 200 ºF	
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
54	<4.0 E-9
59	<2.0 E-8
66	<2.8 E-8
72	<3.4 E-8

Test 9, 200 ºF	
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
48	<3.0 E-8
53	2.6 E-8
59	2.4 E-9

Test 10, 200 ºF	
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
48	<1.3 E-9
54	<2.2 E-8
60	<4.0 E-9
66	<2.0 E-8

Test 11, 200 ºF	
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
48	7.3 E-9
54	<2.6 E-8
59	2.7 E-7 *
66	<2.4 E-8

Test 27, 200 ºF	
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
35	<4.0 E-9
41	<1.6 E-8
48	<2.6 E-8
54	<2.8 E-8

Test 35, 200 ºF	
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
48	2.8 E-8
54	<1.8 E-8
60	<2.6 E-8
66	<2.6 E-8

* Test 11 – failed bag test at 59 months. Each O-ring ok individually.

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 36, 200 ºF	
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
48	3.7 E-9
53	<3.2 E-8
60	<6.0 E-9
66	<2.2 E-8

Test 37, 200 ºF	
2E5Rad /72 min	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	(invalid)
8	1.9 E-8
11	<8.0 E-9
24	<1.8 E-8
32	<2.2 E-8
36	<1.0 E-8
41	<3.0 E-8
47	<1.3 E-9
53	<3.6 E-8
60	<6.0 E-9
66	<2.0 E-8

Test 40, 200 ºF	
2E5Rad /72 min	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	7.7 E-9
6	1.5 E-8
12	<1.0 E-8
28	<2.6 E-8
35	<7.0 E-10
42	1.6 E-8
48	4.8 E-7 *
53	<1.2 E-8
60	<3.4 E-8

* Test 40 – failed bag test at 48 months. Both O-rings ok. Later traced to leak at hose connection.

Test 41, 200 ºF	
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	<2.0 E-8
48	2.2 E-8
53	<3.4 E-8
59	<1.4 E-8
65	<2.0 E-8

Test 42, 200 ºF	
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
48	<1.2 E-8
53	<2.8 E-8
60	<1.2 E-8
65	<1.6 E-8

Test 43, 200 ºF	
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
48	<1.3 E-9
53	<3.6 E-8
59	<4.0 E-9

Test 44, 200 ºF	
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
48	<1.6 E-8
54	<2.4 E-8
60	<1.4 E-8
66	<2.0 E-8

Test 53, 200 ºF	
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
48	<2.2 E-8
54	<3.2 E-8
60	<3.6 E-8
66	<2.0 E-8

Test 54, 200 ºF	
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
48	<2.6 E-8
54	<1.8 E-8
59	<2.6 E-8
66	<1.9 E-8

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 55, 200 ºF	
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	1.1 E-8
42	3.7 E-8
48	3.3 E-7 *
53	<3.0 E-8
59	<1.0 E-8
65	<1.6 E-8

Test 56, 200 ºF	
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	<2.4 E-8
48	<1.0 E-8
53	<2.2 E-8
60	<3.2 E-8
66	<1.8 E-8

Test 57, 200 ºF	
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
47	<1.3 E-9
53	<2.6 E-8
59	<6.0 E-9
66	<1.8 E-8

* Test 55 – failed bag test at 48 months. Both O-rings ok. Later traced to leak at hose connection.

Test 4, 300 ºF	
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
52	<1.2 E-8
55	<3.6 E-8
57	6.4 E-8
59	<2.8 E-8
62	<2.8 E-8
66	<2.0 E-8
68	<2.6 E-8
71	<2.6 E-8
74	<1.5 E-8

Test 7, 300 ºF	
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
51	1.9 E-7
53	2.4 E-7 *
57	<2.2 E-8
59	8.2 E-8
63	<3.6 E-8
66	<2.8 E-8
68	<2.8 E-8
71	<1.5 E-8

Test 8, 300 ºF	
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
52	1.1 E-7
55	<4.2 E-8
56	>9 E-5 **
59	<2.8 E-8
62	<1.6 E-8
66	<2.8 E-8
68	>9 E-5 **

* Test 7 – failed bag test at 53 months. Both O-rings ok. Later traced to leak at hose connection.
** Test 8 – failed bag test at 56 months. Both O-rings ok. Later traced to leak at hose connection. Both O-rings failed at 68 months.

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 18, 300 ºF	
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	*
42	9.4 E-8
46	1.3 E-8
47	<2.6 E-8
50	<3.6 E-8
53	<1.0 E-8
57	<3.8 E-8
59	8.8 E-9
63	1.8 E-8

Test 20, 300 ºF	
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
48	<2.0 E-8
51	8.2 E-8
55	3.7 E-8
58	<1.6 E-8
60	<2.4 E-8
63	<2.2 E-8
65	2.8 E-8
69	<2.0 E-8
71	<1.7 E-8

Test 30, 300 ºF	
2E5Rad /72 min	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	(invalid)
6	5.6 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
46	5.6 E-7 **
48	3.6 E-8
51	2.4 E-8
54	3.6 E-8
57	7.0 E-8
59	4.4 E-8
63	<5.2 E-8

* Test 18 – Overall fixture test at 39 months not successful, but each O-ring passed individually
** Test 30 – Inner O-ring failed at 46 months. Subsequent tests on outer O-ring only. Rates uncorrected at 48 – 59 months.

Test 33, 300 ºF	
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	*
45	4.7 E-8
48	1.7 E-6 *
50	2.8 E-8
53	2.4 E-8
57	2.4 E-8
59	3.0 E-8
63	8.8 E-8
65	8.2 E-7 *

Test 51, 300 ºF	
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
43	6.5 E-8
45	3.9 E-6 **
48	<1.8 E-8
51	<2.4 E-8
54	4.6 E-8
57	<3.2 E-8
60	<2.6 E-8
63	<1.6 E-8

Test 52, 300 ºF	
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 ***
39	<4.0 E-9 ***
42	<7.2 E-8 ***
45	<2.6 E-8
48	<3.8 E-8
51	<1.2 E-8
55	<4.6 E-8
57	<2.8 E-8
60	<4.2 E-8
61	<1.6 E-8

* Test 33 – Overall fixture test at 42 months not successful, but each O-ring passed individually. Inner O-ring failed at 48 months. Outer O-ring only, rates uncorrected at 50 – 63 months. Outer O-ring failed at 65 months.
** Test 51 – failed bag test at 45 months. Both O-rings ok. Later traced to leak at hose connection.
*** Test 52 – outer O-ring apparently failed at 36 months. Test of inner O-ring only at 39 months. Test both O-rings at 42 months – both ok. Subsequent tests on both O-rings.

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 61, 300 ºF		Test 14W, 270 ºF		Test 21W, 270 ºF	
No rad.		No rad.		No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)	Time at Temp (months)	Leak Rate (std cc He/sec)	Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	(invalid)	Baseline	<1.6 E-8	Baseline	<1.6 E-8
6	1.8 E-8	5	<2.6 E-8	6	<2.4 E-8
11	<1.0 E-8	12	<2.8 E-8	12	<3.0 E-8
23	<1.6 E-8				
30	<2.0 E-8				
36	1.8 E-8				
42	4.7 E-8				
47	2.4 E-7 *				
51	<2.2 E-8				
53	<1.2 E-8				
57	<2.0 E-8				
60	<2.8 E-8				
62	<2.6 E-8				
65	<1.6 E-8				

* Test 61 – failed bag test at 47 months. Both O-rings ok. Later traced to leak at hose connection.

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 13H (GLT-S), 200 ºF	
No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)
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
14	<1.0E-08
20	4.3 E-7 *
25	<1.6 E-8
30	<1.8 E-8
35	<3.8 E-8

Test 15H (GLT-S), 200 ºF	
No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)
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	1.5E-08
20	2.4 E-7 **
26	<1.0 E-8
30	<1.6 E-8
36	<4.2 E-8

Test 16H (GLT-S), 250 ºF	
No rad..	
Time at Temp (months)	Leak Rate (std cc He/sec)
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	1.5E-07
12	2.4 E-8
14	3.8 E-7 ***
15	<1.2 E-8
16	>9 E-5 ***
22	<1.8 E-8
30	1.6 E-8

* Test 13H – failed bag test at 20 months. Both O-rings ok. Later traced to leak at hose connection.
** Test 15H – failed bag test at 20 months. Both O-rings ok. Later traced to leak at hose connection.
*** Test 16H – failed bag test at 14 and 16 months. Both O-rings ok. Later traced to leak at hose connection.

Table 4. (cont.) Room temperature leak rate data for fixtures (for fixtures in test since last status report)

Test 22H (GLT-S), 250 ºF	
No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	<9.1 E-09
0.4	<1.0 E-08
0.9	3.8 E-07
1.2	9.0 E-09
1.7	<2.9 E-09
2.1	2.0 E-09
2.6	2.6 E-09
3.1	<1.0 E-09
3.7	8.8 E-10
4.2	3.7 E-09
4.8	<8.0 E-09
5.4	<9.6 E-10
5.8	<8.0 E-09
6.4	<9.1 E-10
6.8	<2.8 E-08
7.3	<1.1 E-09
8.0	<7.3 E-10
8.6	<2.0 E-08
9.3	<2.6 E-08
11	<1.8 E-08
12	<2.0 E-08
13	3.9 E-08
14	2.1 E-5 *
16	3.2 E-8
17	<3.0 E-8
18	<6.6 E-8
24	<1.4 E-8
30	<2.6 E-8

Test 29H (GLT-S), 300 ºF	
No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	<8.2 E-09
0.5	1.6 E-08
1.1	<8.1 E-09
1.7	6.2 E-09
2.0	<1.0 E-09
2.5	1.5 E-09
3.2	<1.1 E-09
3.8	2.2 E-08
4.2	4.2 E-09
4.9	9.9 E-09
5.5	<9.6 E-10
5.9	<8.0 E-09
6.2	<9.1 E-10
6.4	<2.4 E-08
6.9	<1.1 E-09
7.5	<6.7 E-10
8.2	<1.8 E-08
8.8	1.2 E-08
9.8	<2.0 E-08
11	2.9 E-08
12	<1. 0E-08
14	1.1 E-7
15	<1.4 E-8
16	<4.0 E-9
17	2.2 E-7 **
18	3.8 E-8
20	<2.6 E-8
23	<2.4 E-8
27	3.9 E-8
30	<2.8 E-8
33	3.4 E-8

Test 34H (GLT-S), 300 ºF	
No rad.	
Time at Temp (months)	Leak Rate (std cc He/sec)
Baseline	<9.5 E-09
0.8	<1.0 E-08
1.2	<2.0 E-09
1.8	<8.0 E-09
2.4	<3.2 E-09
2.7	<1.0 E-09
3.2	2.2 E-09
3.8	<1.1 E-09
4.5	8.8 E-10
4.9	2.4 E-09
5.6	<1.0 E-08
6.2	<9.5 E-09
6.5	<1.2 E-08
7.1	<9.1 E-10
7.6	<2.4 E-08
8.1	<1.0 E-09
8.7	<6.8 E-10
9.3	<1.6 E-08
10	<1.6 E-08
11	<1.6 E-08
12	<3.0 E-08
13	<1.2 E-09
14	<1.8 E-8
16	<1.4 E-8
17	5.2 E-9
18	3.2 E-5 ***
21	<1.7 E-8
24	<1.4 E-8
27	<3.2 E-8
30	<3.0 E-8
33	<2.2 E-8
36	<1.4 E-8

* Test 22H – outer O-ring apparently failed at 14 months. Test of inner O-ring only 16 - 18 months. Test both O-rings at 24 months – both ok.

** Test 29H - failed bag test at 17 months. Both O-rings ok. Later traced to leak at hose connection.

*** Test 34H – leak at hose connection at 18 months. Subsequent tests ok.

Table 5. 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 ^{o}F)					30 days	62% / 59%
29 (283 days at 200 ^{o}F)					30 days	30% / 18%
38 (473 days at 300 ^{o}F)	<30 min.	85% / 87%				
39 (456 days at 300 ^{o}F)	<30 min.	77% / 81%				
45 (291 days at 300 ^{o}F)					30 days	60% / 71%
46 (493 days at 300 ^{o}F)	<30 min.	76% / 75%				
47 (394 days at 300 ^{o}F)	1 hour	80% / 81%	5 days	77% / 73%	34 days	68% / 72%
48 (490 days at 300 ^{o}F)	<30 min.	84% / 84%				
50 (265 days at 300 ^{o}F)					30 days	42% / 38%
60 (454 days at 300 ^{o}F)	<30 min.	88% / 89%				
62 (282 days at 300 ^{o}F)					30 days	50% / 54%
GLT O-Ring Fixtures removed after failing room temperature leak test						
8 (2082 days at 300 ^{o}F)	15 minutes	90% / 92%	13 days	94% / 91%	30 days	94% / 89%
12 (1020 days at 300 ^{o}F)	7 minutes	82% / 70%	14 days	75% / 55%	30 days	74% / 49%
14D (45 days at 400 ^{o}F)	21 minutes	51% / 77%	9 days	54% / 74%	85 days	45% / 66%
18D (497 days at 350 ^{o}F)	23 minutes	91% / 96%	14 days	93% / 97%	30 days	92% / 97%
19D (594 days at 350 ^{o}F)	13 minutes	95% / 94%	14 days	98% / 98%	30 days	97% / 97%
21D (27 days at 400 ^{o}F)	27 minutes	66% / 77%	9 days	58% / 69%	80 days	53% / 66%
23D (12 days at 450 ^{o}F)	21 minutes	65% / 70%	14 days	53% / 63%	90 days	54% / 59%
26 (1410 days at 300 ^{o}F)	10 minutes	90% / 91%	14 days	88% / 89%	30 days	88% / 88%
31 (1292 days at 300 ^{o}F)	15 minutes	84% / 78%	14 days	80% / 67%	31 days	78% / 65%
32 (1352 days at 300 ^{o}F)	14 minutes	93% / 83%	14 days	90% / 73%	31 days	89% / 71%
49 (1360 days at 300 ^{o}F)	14 minutes	84% / 81%	14 days	82% / 80%	30 days	81% / 79%
GLT O-Ring Fixtures removed for other reasons						
28 (630 days at 200 ^{o}F)	4 hours	68% / 62%	10 days	31% / 28%	230 days	28% / 24%
62-2007 (~6 months at 300 ^{o}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 ºF)	10 minutes	84% / 91%	11 days	80% / 88%	26 days	80% /88%
38H (358 days at 350 ºF)	20 minutes	92% / 92%	14 days	90% /88%	30 days	88% /87%
39H (114 days at 350 ºF)	15 minutes	78% /90%	11 days	74% /89%	26 days	72% /88%
45H (99 days at 400 ºF)	12 minutes	93% /93%	11 days	91% /92%	26 days	91% /91%
50H (281 days at 400 ºF)	14 minutes	95% /82%	14 days	93% /76%	30 days	93% /76%
58H (75 days at 400 ºF)	10 minutes	83% /87%	11 days	81% /84%	26 days	78% /84%
60H (50 days at 400 ºF)	7 minutes	84% /93%	11 days	80% /90%	26 days	79% /89%
62H (50 days at 400 ºF)	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.

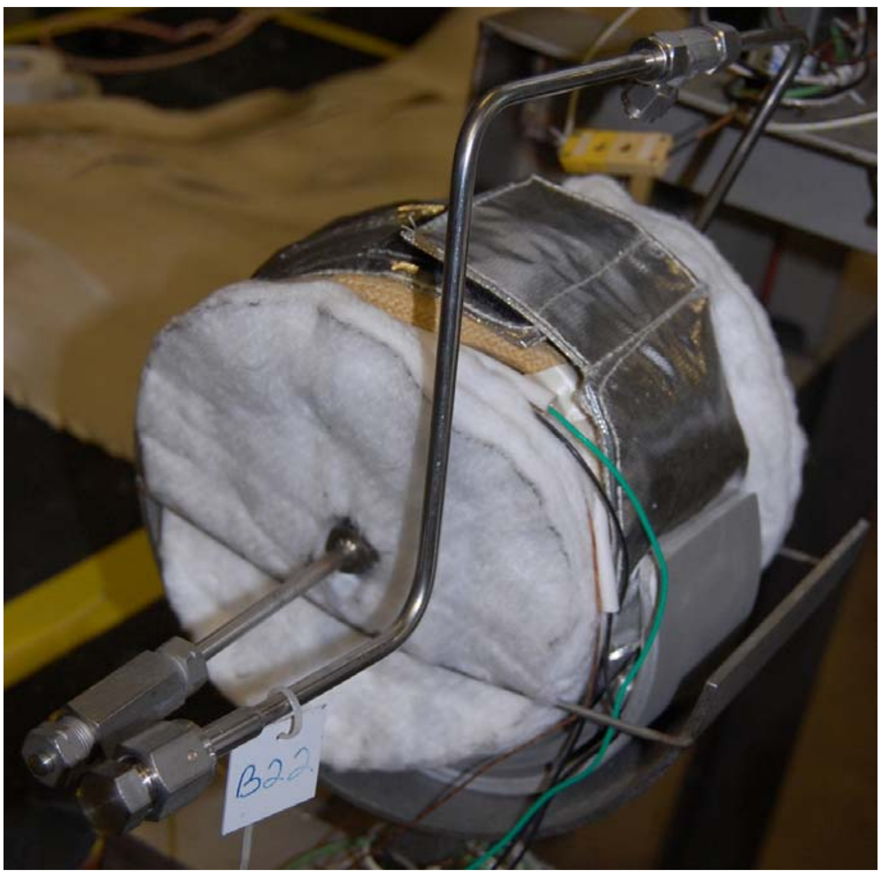
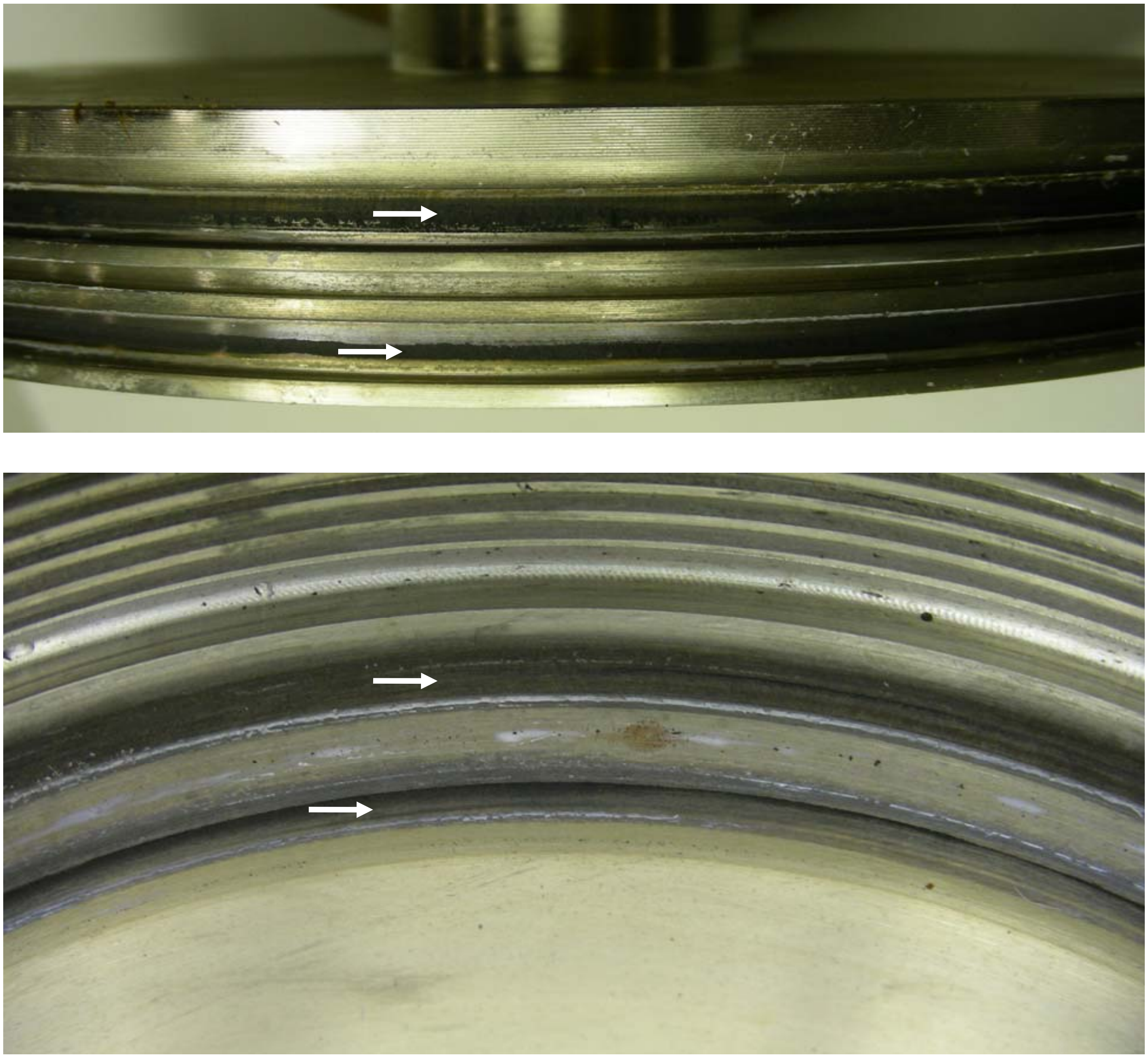


Figure 2. Assembled mock-up PCV.



(a)

(b)

Figure 3. Black stain on fixture 8 plug (a) and body (b) following disassembly.

Figure 4. Fixture 8 outer O-ring after removal with cracking and pullout along the surface facing the bottom of the groove.

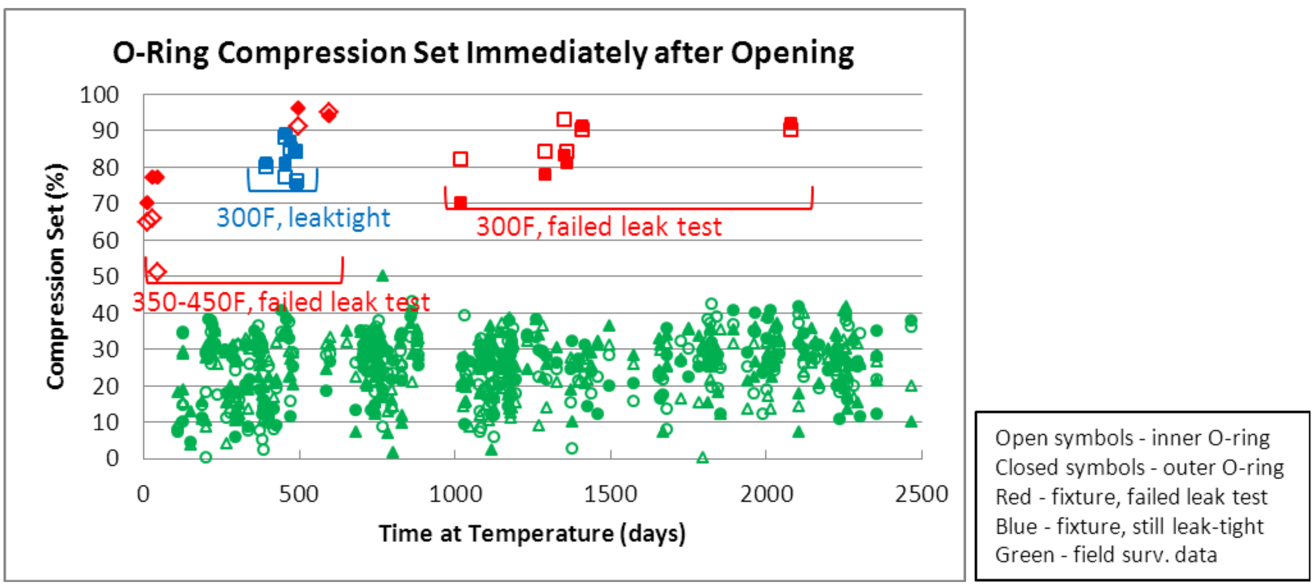


Figure 5. Compression set data for GLT O-rings based on measurements taken immediately after opening vessel (typically 30 minutes or less). The red symbols represent test fixtures aged at 300 °F or higher that failed to remain leaktight at room temperature. The blue symbols represent test fixtures aged at 300 °F removed from test due to difficulty with elevated temperature leak testing but remained leaktight at room temperature. The green symbols are from field surveillance PCV and SCV O-rings (presumed leaktight).

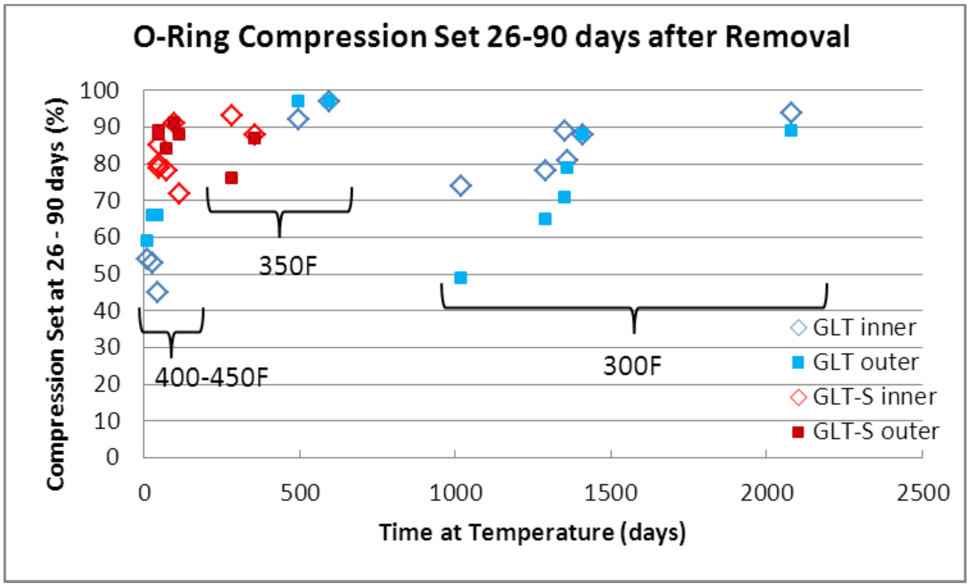


Figure 6. Compression set of GLT and GLT-S O-rings removed from test fixtures which failed to remain leaktight at room temperature. These results are based on re-measurements taken 26 – 90 days after opening the fixture.

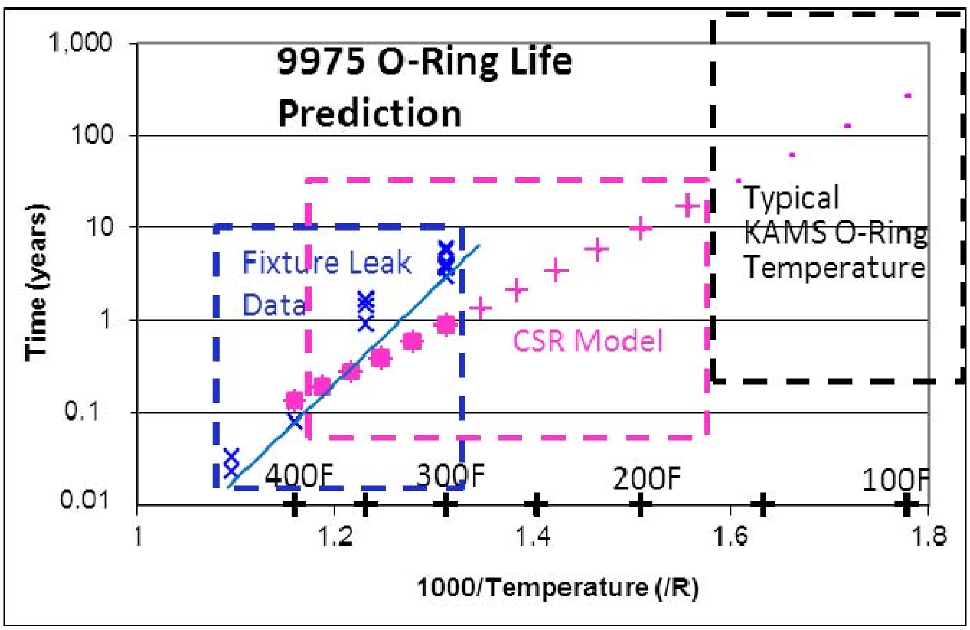


Figure 7 Leakage-based service life data for GLT O-rings (from fixtures with room temperature leak rate failures) compared to life predictions from GLT O-ring compression stress relaxation (CSR) data. The CSR data are distinguished by whether the sample reached a failure condition (90% loss of force, solid symbols), or whether the sample data were extrapolated to estimate a failure condition (+ symbols).

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