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

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Ninth 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. One approach has been to periodically evaluate the leak performance of O-rings being aged in mock-up 9975 Primary Containment Vessels (PCVs) at elevated temperatures. Other methods such as compression-stress relaxation (CSR) tests and field surveillance are also on-going to evaluate O-ring behavior. Seventy tests using PCV mock-ups 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 continue to meet the leak-tightness criterion defined in ANSI standard N14.5-97. Due to material substitution, 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 earliest 300 °F GLT O-ring fixture failure was observed at 34 months. 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 72 - 96 months, which bounds O-ring temperatures anticipated during storage in K-Area Complex (KAC). Based on expectations that the 200 ºF fixtures will remain leak-tight for a significant period yet to come, 2 additional fixtures began aging in 2011 at 270 ºF, with hopes that they may reach a failure condition before the 200 ºF fixtures, thus providing additional time to failure data.

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 54 - 57 months.

No additional O-ring failures have been observed since the last interim report was issued. Aging and periodic leak testing will continue for the remaining PCV fixtures. Additional irradiation of several fixtures is recommended to maintain a balance between thermal and radiation exposures similar to that experienced in storage, and to show the degree of consistency of radiation response between GLT and GLT-S O-rings.

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 [4] 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 5. 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 were initially 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 vendor's 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. Fewer fixtures were used for this alternate O-ring material since it is expected they will demonstrate the same parametric variations as the GLT O-rings. 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) [6]. 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 to verify leak-tightness to 1E-7 ref·cc/sec air or better. 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 (lasting approximately 240 hrs) or a faster rate of ~1.7E5 rad/hr (lasting 72 minutes), asViton® and other elastomers/polymers are known to be sensitive to the dose rate. After irradiation, the fixture was leak tested again while at room temperature to ensure that irradiation alone did not affect leak-tightness, and then 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 [1, 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 [4]. Initially, fixtures were also tested at their conditioning temperature. These additional tests were discontinued, as discussed previously [5].

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 [4]. 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, 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 [7].

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 7), 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. The time to permeation ranged from 4-75 minutes?

Results

PCV fixtures have been assembled and aged to identify the time to failure of GLT O-rings (70 tests), and GLT-S O-rings (14 tests). This report summarizes results for these fixtures through July 10, 2014.

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 72 to 96 months (at the time of their last leak test). Two fixtures began conditioning at 270 ºF in 2011. They have remained leak-tight at room temperature, with total time at temperature of 35 months (at the time of their last leak test). Eight 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. In one of these fixtures (fixture 30) the inner O-ring had failed, but the outer O-ring remained leak-tight. Three of these fixtures were opened to examine the O-rings, while the remaining 5 fixtures are being maintained as-is for potential future test initiatives. GLT O-rings in the remaining fixtures aging at 300 ºF 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. Leak rate histories can be found in Table 4 for fixtures in test since the last status report [8].

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 54 to 57 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 4 for fixtures in test since the last status report [8].

Compression set values for all the opened fixtures have been calculated per ASTM D395 Method B and are summarized in Table 5.

Discussion

As noted in the previous interim report [8], sufficient data are available to compare the time to failure for GLT and GLT-S O-rings at 3 temperatures. At 400 ºF, most of the GLT-S O-ring fixtures remained leak-tight longer than the GLT O-ring fixtures. At 350 ºF, the trend was reversed, with 3 of the 4 GLT O-rings in two fixtures remaining leak-tight longer than the GLT-S O-ring fixtures. At 300 ºF, both GLT-S O-ring fixtures remain leak-tight after 45 months, while 5 GLT O-ring fixtures had failed by this time. With this varying trend, it is premature to conclude one material would perform better than the other, especially at the lower temperatures typical of storage service. This conclusion remains unchanged from the previous report since no additional leakage failures have occurred.

It is noted that GLT and GLT-S O-ring leak failures have occurred at 400 °F within 1 month and at 350 °F within one year In addition, GLT O-ring leak failures have occurred at 300 °F within 3 years. These temperatures are at or below the typical “maximum” service temperature often quoted for Viton/FKM-type fluoroelastomers (400 °F). This highlights the limitation of such values for design purposes, as the time at temperature and failure criterion truly dictate service life.

A bounding steady state O-ring temperature of 199 ºF has been calculated for an ambient temperature of 137 ºF and 19 watts payload [10]. Reference 11 reports the maximum ambient temperature actually recorded (at that time) was 104 ºF, although temperatures are more typically 90 ºF or less. For the more realistic bounding ambient temperature of 104 ºF, the corresponding O-ring temperature would be expected to drop to ~166 ºF. Lower internal heat loads and ambient temperatures more representative of typical storage conditions would lead to further reductions in the O-ring temperature. Even if bounding temperatures are reached, they are not sustained for chronic periods

The times at temperature for the fixtures to fail the room temperature leak test are shown graphically in Figure 3. In addition, the times at temperature for the fixtures still in test are also shown. A trend line is shown that provides a lower bound to the failure data to illustrate a potential extrapolation of the observed behavior to temperatures below 300 ºF, although the degree of scatter in the data would suggest significant uncertainty for extrapolations over a large temperature range. Nevertheless, this trend line suggests the possibility that O-rings aging at 200 ºF might maintain a leak-tight seal for approximately 75 years, and O-rings at KAC storage temperatures have the potential for even greater service life.

The fixtures which were irradiated received a bounding 10 year dose (2E5 rads), based on the initial approved storage period. While thermal aging is expected to dominate O-ring behavior due to the low dose rates involved in service, the approved storage period has since been increased to 15 years, and further extensions are possible. In addition, there is an inherent imbalance between the bounding irradiation dose and the O-ring aging conditions. A bounding O-ring temperature of approximately 200 ºF was originally identified. Given that actual O-ring temperatures in storage are typically ~166 ºF or less, aging at 200 ºF and above represents a significant acceleration of thermal aging. The aging model based on CSR data indicates O-ring degradation will occur ~2.7 times faster at 200 ºF than at 166 ºF, meaning that 8 years aging at 200 ºF represents about 22 years aging at 166 ºF. This same acceleration was not applied to the radiation dose. To maintain balance between these two degradation sources, it is recommended that some of the remaining fixtures which were initially irradiated receive further irradiation, as follows:

- 3 of the 200 ºF fixtures should receive an additional 2 E5 rads (at the high dose rate) following their next scheduled leak test. At that time, they will have approximately 8 years aging time (at 200 ºF) which corresponds to approximately 22 years aging at ~166 ºF. These same 3 fixtures should continue to receive periodic irradiation to maintain this ratio of approximately 2E5 rads per 4 years at 200 ºF. These exposures do not ideally duplicate simultaneous radiation/thermal aging in service, but are intended to maintain a balance between degradation mechanisms.
- 3 of the 200 ºF fixtures should receive additional periodic irradiations to maintain a ratio of approximately half of the above bounding dose (1 E5 rads) per 4 years at 200 ºF, to provide a more realistic dose rate. The first of these irradiations would follow their next scheduled leak test.

- 1 of the retired (but unopened) 300 ºF fixtures should receive additional irradiation to a 50 year dose (1 E6 rads total), a 100 year dose (2 E6 rads total) and a 150 year dose (3 E6 rads total). 5 years aging at 300 ºF corresponds to approximately 155 years aging at 166 ºF. This fixture should be leak tested before each irradiation, and cycled to 300 ºF and leak tested after each irradiation.

Each of the above irradiations will be performed on fixtures containing Viton GLT O-rings, since the GLT-S O-rings aging at these temperatures were not irradiated. This is mainly because at the time of GLT-S introduction into the test program, radiation was considered to be of less significance than thermal degradation. To better verify whether GLT-S O-rings respond comparably to GLT O-rings, the following steps are recommended:

- Install 2 GLT O-rings on a PCV lid (~18% stretch but no compression) and irradiate in incremental doses at the highest dose rate available until cracking is observed. Repeat with 2 GLT-S O-rings. Target doses have not been selected, but doses of 10, 50, 75, 100, 150 and 200 Mrad are suggested.
- Install 2 GLT O-rings on a PCV lid and torque onto a fixture (~18% stretch plus compression) and irradiate to the dose identified above. Test if the fixture remains leak-tight. If so, irradiate again to 2x, 5x and 10x the above dose, or until the fixture leaks (whichever comes first). Repeat for 2 GLT-S O-rings. These tests will not address possible variation in dose rate sensitivity between the two compounds, but such variation is expected to be minor based on similar polymer compositions.

Conclusions

High temperature aging continues for 23 GLT O-ring fixtures at 200 – 270 ºF. Room temperature leak test failures have been experienced by one or both O-rings 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 72 - 96 months, which is more representative of (but still bounding to) O-ring temperatures during storage in KAC. The maximum O-ring temperature expected in the KAC is ~ 166 °F, based on a transient peak ambient temperature of 104 °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 54 - 57 months.

Aging and periodic leak testing will continue for the remaining fixtures. Additional irradiation of several fixtures is recommended to maintain a balance between thermal and radiation exposures similar to that experienced in storage, and to account for storage periods longer than the initial 10-year period. Tests are also recommended to show the degree of consistency of radiation response between GLT and GLT-S O-rings.

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] 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.

[5] 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

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

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

[8] SRNL-TR-2013-00151, "Eighth Interim Status Report: Model 9975 PCV O-Ring Fixture Long-Term Leak Performance", W. L. Daugherty, August 2013.

[9] SRNS-TR-2008-00290, Rev. 0, "Summary and Matrix 9975 Shipping Package Qualification Program for Extended Storage of Plutonium in the K Area Complex", J. A. Radder, Savannah River Nuclear Solutions, Aiken, SC, November 2008.

[10] S-CLC-K-00194, Rev. 2, "Integrated Thermal Analysis of the 9975 Shipping Packages for KAMS Phase 6", J. K. Norkus, March 16, 2011.

[11] OBU-NMM-2004-00060, "KAMS T-1 Temperature Readings", S. M. Herlihy, March 25, 2004.

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

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 [9], and the corresponding PCV O-ring temperature is 199 °F [10].
	300 °F (149 °C)	The maximum allowable temperature for the PCV O-rings for continuous operation is 300 °F [6].
	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 (high) 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. (lower dose rate)
	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 [12].
	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 [12].
	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	1902 – no fail
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 since 2011 (for fixtures in test since last status report)

Test 1, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	60	<3.0 E-8
9/19/2011	66	<3.0 E-8
3/19/2012	72	<2.0 E-9
10/8/2012	78	1.3 E-8
7/9/2013	85	5.3 E-9
12/3/13	90	1.3 E-8
6/10/14	96	2.4 E-8

Test 3, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	59	<2.8 E-8
8/1/2011	65	<1.8 E-8
2/22/2012	72	<1.6 E-8
9/5/2012	80	<1.0 E-8
5/14/2013	84	5.9 E-9
11/12/13	90	5.8 E-9
5/13/14	96	1.3 E-8

Test 5, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/14/2011	59	<2.4 E-8
10/24/2011	66	<2.6 E-8
4/30/2012	71	1.0 E-7
11/15/2012	78	<1.6 E-9
7/25/13	84	8.7 E-9
1/22/14	90	2.7 E-8

Test 6, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/14/2011	59	<2.0 E-8
10/24/2011	66	<2.8 E-8
5/1/2012	72	<3.4 E-8
11/15/2012	78	9.1 E-9
7/9/2013	84	1.5 E-8
1/7/14	90	<2.5 E-9
7/8/14	96	3.8E-8

Test 9, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	53	2.6 E-8
1/24/2012	59	2.4 E-9
8/15/2012	66	<1.2 E-8
4/9/2013	72	<2.4 E-9
10/8/13	78	2.8 E-9
4/8/14	84	6.0 E-9

Test 10, 200 ºF		
2E5Rad /240 hr		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	54	<2.2 E-8
8/16/2011	60	<4.0 E-9
2/22/2012	66	<2.0 E-8
9/10/2012	72	1.6 E-8
5/14/2013	78	<1.7 E-9
11/12/13	84	1.2 E-9
5/13/14	90	8.6 E-9

Test 11, 200 ºF		
1.4E5 Rad /479 hr		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	54	<2.6 E-8
8/16/2011	59	2.7 E-7 *
3/19/2012	66	<2.4 E-8
10/8/2012	72	2.7 E-9
6/4/2013	78	5.5 E-9
12/3/13	84	7.8 E-9
5/3/14	89	8.6 E-9

Test 27, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	41	<1.6 E-8
10/24/2011	48	<2.6 E-8
5/30/2012	54	<2.8 E-8
12/11/2012	61	5.8 E-9
8/13/13	67	8.2 E-9
1/22/14	72	5.4 E-9

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

Table 4. (cont) Room temperature leak rate data since 2011 (for fixtures in test since last status report)

Test 35, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	54	<1.8 E-8
10/24/2011	60	<2.6 E-8
4/30/2012	66	<2.6 E-8
11/15/2012	73	<1.6 E-9
7/9/2013	78	1.7 E-8
1/7/14	84	9.8 E-8
7/8/14	90	3.8 E-8

Test 36, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	53	<3.2 E-8
8/16/2011	60	<6.0 E-9
3/19/2012	66	<2.2 E-8
10/8/2012	72	<1.9 E-9
6/4/2013	78	<1.9 E-9
12/2/13	84	7.8 E-9
6/10/14	90	3.3 E-9

Test 37, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	53	<3.6 E-8
8/16/2011	60	<6.0 E-9
3/19/2012	66	<2.0 E-8
10/8/2012	72	2.7 E-8
6/4/2013	78	<1.9 E-9
12/3/13	84	2.6 E-9
6/10/14	90	4.3 E-8

Test 40, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	53	<1.2 E-8
1/24/2012	60	<3.4 E-8
8/15/2012	66	<1.4 E-8
4/9/2013	72	2.2 E-9
10/8/13	78	2.8 E-9
4/8/14	84	1.8 E-8

Test 41, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	53	<3.4 E-8
8/1/2011	59	<1.4 E-8
2/22/2012	65	<2.6 E-8
9/10/2012	72	<1.6 E-8
5/14/2013	78	<1.7 E-9
11/12/13	84	<2.9 E-9
5/13/14	90	3.9 E-9

Test 42, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	53	<2.8 E-8
8/1/2011	60	<1.2 E-8
2/22/2012	65	<1.6 E-8
9/6/2012	72	<1.2 E-8
5/14/2013	78	7.1 E-9
11/13/13	84	1.7 E-8
5/13/14	90	2.9 E-8

Test 43, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	53	<3.6 E-8
8/16/2011	59	<4.0 E-9
3/19/2012	66	<9.8 E-10
10/8/2012	72	2.7 E-9
6/4/2013	78	<1.9 E-9
12/3/13	84	1.6 E-8
6/18/14	90	6.4 E-9

Test 44, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	54	<2.4 E-8
8/1/2011	60	<1.4 E-8
2/22/2012	66	<2.0 E-8
9/6/2012	72	<1.0 E-8
5/14/2013	78	<1.7 E-9
11/12/13	84	5.8E-9
5/13/14	90	1.2 E-8

Table 4. (cont) Room temperature leak rate data since 2011 (for fixtures in test since last status report)

Test 53, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	54	<3.2 E-8
8/1/2011	60	<3.6 E-8
2/22/2012	66	<2.0 E-8
9/10/2012	72	<2.2 E-8
5/14/2013	78	1.2 E-8
11/12/13	84	<2.0 E-9
5/13/14	90	5.5 E-8

Test 54, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	54	<1.8 E-8
8/1/2011	59	<2.6 E-8
2/22/2012	66	<1.9 E-8
9/10/2012	72	<2.0 E-8
5/14/2013	78	1.2 E-8
11/12/13	84	<2.0 E-9
5/13/14	90	<2.7 E-9

Test 55, 200 ºF		
2E5Rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/1/2011	53	<3.0 E-8
8/1/2011	59	<1.0 E-8
2/22/2012	65	<1.6 E-8
9/10/2012	72	<1.6 E-8
5/14/2013	78	2.4 E-9
11/12/13	84	2.9 E-9
5/13/14	90	4.0 E-8

Test 56, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	53	<2.2 E-8
10/24/2011	60	<3.2 E-8
4/30/2012	66	<1.8 E-8
11/15/2012	72	<1.6 E-9
7/9/2013	78	1.1 E-8
1/7/14	84	7.4 E-8
7/8/14	90	1.6 E-8

Test 57, 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
2/15/2011	53	<2.6 E-8
8/16/2011	59	<6.0 E-9
3/20/2012	66	<1.8 E-8
10/8/2012	72	<1.9 E-9
6/4/2013	78	2.8 E-9
12/3/13	84	2.6 E-9
6/18/14	90	1.0 E-8

Test 14W, 270 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/18/2011	Baseline	<1.6 E-8
10/24/2011	5	<2.6 E-8
5/30/2012	12	<2.8 E-8
8/13/13	25	8.2 E-9
7/9/14	35	1.2 E-7

Test 21W, 270 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/18/2011	Baseline	<1.6 E-8
10/24/2011	6	<2.4 E-8
5/30/2012	12	<3.0 E-8
8/13/13	25	5.5 E-9
7/8/14	35	1.6 E-8

Table 4. (cont) Room temperature leak rate data since 2011 (for fixtures in test since last status report)

Test 13H (GLT-S), 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	25	<1.6 E-8
11/28/2011	30	<1.8 E-8
5/29/2012	35	<3.8 E-8
12/11/2012	42	5.8 E-9
8/13/13	48	<1.9 E-9
2/20/14	54	1.6 E-8

Test 15H (GLT-S), 200 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	26	<1.0 E-8
11/28/2011	30	<1.6 E-8
5/29/2012	36	<4.2 E-8
12/11/2012	42	5.8 E-9
8/13/13	48	5.5 E-9
1/22/14	54	<1.9 E-9

Test 16H (GLT-S), 250 ºF		
No rad..		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	22	<1.8 E-8
3/20/2012	30	1.6 E-8
10/8/2012	36	1.3 E-8
6/4/2013	41	<1.9 E-9
12/3/13	48	7.8 E-9
6/10/14	54	3.3 E-9

Test 22H (GLT-S), 250 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/27/2011	24	<1.4 E-8
1/24/2012	30	<2.6 E-8
8/15/2012	37	<1.1 E-8
4/9/2013	42	5.4 E-8
10/8/13	48	1.1 E-8
4/8/14	54	9.0 E-9

Test 29H (GLT-S), 300 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	20	<2.6 E-8
6/8/2011	23	<2.4 E-8
10/18/2011	27	3.9 E-8
1/10/2012	30	<2.8 E-8
4/30/2012	33	3.4 E-8
8/15/2012	36	<1.1 E-8
11/15/2012	39	9.1 E-9
4/9/2013	42	9.9 E-9
7/9/2013	45	1.3 E-8
10/8/13	48	8.3 E-9
1/7/14	51	2.7 E-8
4/8/14	54	1.5 E-8
7/8/14	57	1.7 E-9

Test 34H (GLT-S), 300 ºF		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/15/2011	21	<1.7 E-8
6/8/2011	24	<1.4 E-8
9/19/2011	27	<3.2 E-8
12/28/2011	30	<3.0 E-8
3/19/2012	33	<2.2 E-8
7/11/2012	36	<1.4 E-8
10/8/2012	39	8.0 E-9
3/5/2013	42	2.3 E-9
6/4/2013	45	5.5 E-9
9/4/13	48	5.7 E-9
12/3/13	52	2.6 E-9
3/10/14	54	1.5 E-8
6/10/14	57	1.3 E-8

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 °F)					30 days	62% / 59%
29 (283 days at 200 °F)					30 days	30% / 18%
38 (473 days at 300 °F)	<30 min.	85% / 87%				
39 (456 days at 300 °F)	<30 min.	77% / 81%				
45 (291 days at 300 °F)					30 days	60% / 71%
46 (493 days at 300 °F)	<30 min.	76% / 75%				
47 (394 days at 300 °F)	1 hour	80% / 81%	5 days	77% / 73%	34 days	68% / 72%
48 (490 days at 300 °F)	<30 min.	84% / 84%				
50 (265 days at 300 °F)					30 days	42% / 38%
60 (454 days at 300 °F)	<30 min.	88% / 89%				
62 (282 days at 300 °F)					30 days	50% / 54%
GLT O-Ring Fixtures removed after failing room temperature leak test						
8 (2082 days at 300 °F)	15 minutes	90% / 92%	13 days	94% / 91%	30 days	94% / 89%
12 (1020 days at 300 °F)	7 minutes	82% / 70%	14 days	75% / 55%	30 days	74% / 49%
14D (45 days at 400 °F)	21 minutes	51% / 77%	9 days	54% / 74%	85 days	45% / 66%
18D (497 days at 350 °F)	23 minutes	91% / 96%	14 days	93% / 97%	30 days	92% / 97%
19D (594 days at 350 °F)	13 minutes	95% / 94%	14 days	98% / 98%	30 days	97% / 97%
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%
26 (1410 days at 300 °F)	10 minutes	90% / 91%	14 days	88% / 89%	30 days	88% / 88%
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%
49 (1360 days at 300 °F)	14 minutes	84% / 81%	14 days	82% / 80%	30 days	81% / 79%
33 (1979 days at 300 °F)	10 minutes	88% / 82%	12 days	84% / 74%	29 days	84% / 73%
GLT O-Ring Fixtures removed for other reasons						
28 (630 days at 200 °F)	4 hours	68% / 62%	10 days	31% / 28%	230 days	28% / 24%
62-2007 (~6 months at 300 °F)	4 hours	66% / 77%	11 days	35% / 35%	230 days	32% / 31%
7 (2167 days at 300 °F)	19 minutes	93% / 84%	12 days	90% / 78%	29 days	90% / 75%
30 (1902 days at 300 °F)	16 minutes	94% / 82%	12 days	90% / 73%	29 days	89% / 71%
52 (1848 days at 300 °F)	12 minutes	92% / 87%	12 days	90% / 80%	29 days	88% / 79%
GLT-S O-Ring Fixtures removed after failing room temperature leak test						
28H (50 days at 400 °F)	10 minutes	84% / 91%	11 days	80% / 88%	26 days	80% /88%
38H (358 days at 350 °F)	20 minutes	92% / 92%	14 days	90% /88%	30 days	88% /87%
39H (114 days at 350 °F)	15 minutes	78% /90%	11 days	74% /89%	26 days	72% /88%
45H (99 days at 400 °F)	12 minutes	93% /93%	11 days	91% /92%	26 days	91% /91%
50H (281 days at 400 °F)	14 minutes	95% /82%	14 days	93% /76%	30 days	93% /76%
58H (75 days at 400 °F)	10 minutes	83% /87%	11 days	81% /84%	26 days	78% /84%
60H (50 days at 400 °F)	7 minutes	84% /93%	11 days	80% /90%	26 days	79% /89%
62H (50 days at 400 °F)	7 minutes	89% /91%	11 days	86% /89%	26 days	85% /89%

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

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

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

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

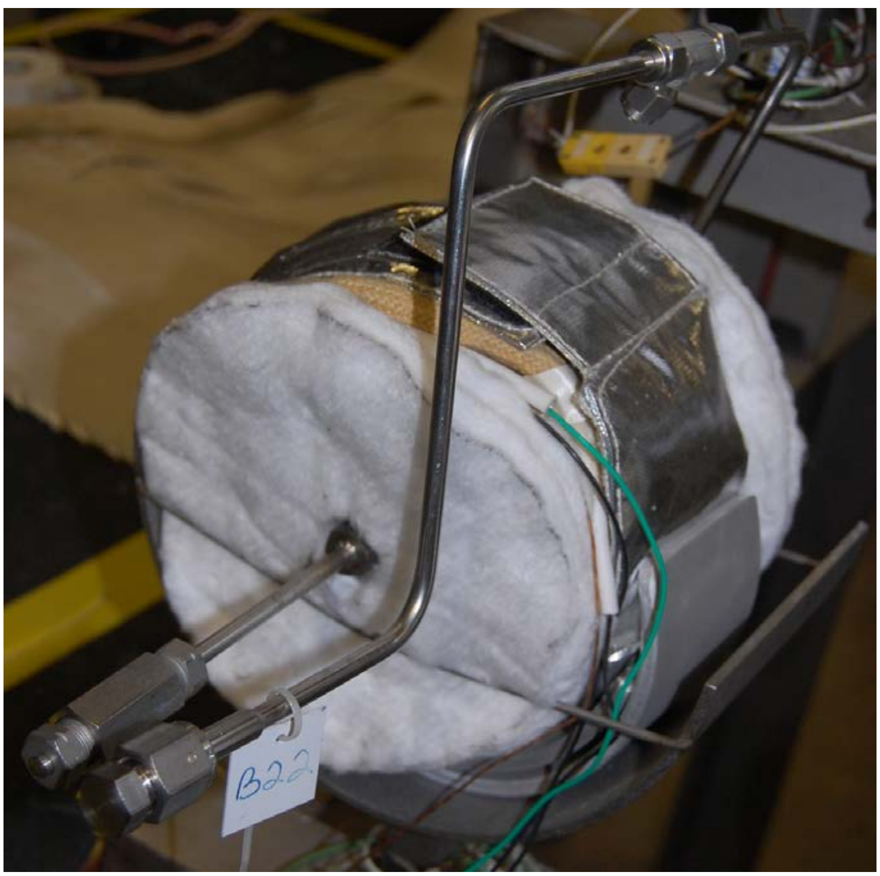

Figure 2. Assembled mock-up PCV.

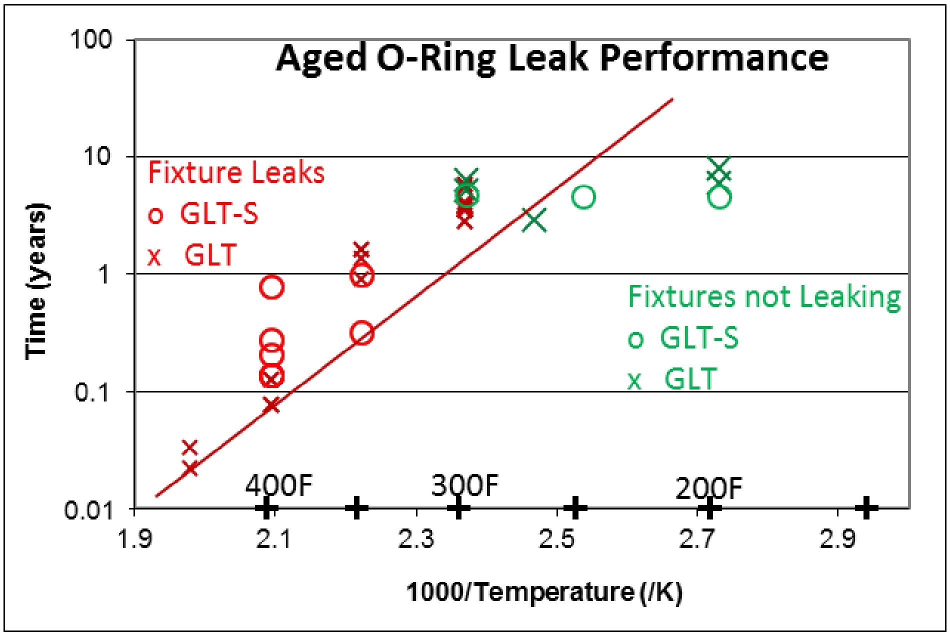

Figure 3. Summary of behavior for fixtures that failed the room temperature leak test and for fixtures still in test. The trend line illustrates a lower bound projection.

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