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Eleventh Interim Status Report: Model 9975 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 temperature. 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 °F. 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 °F.

High temperature aging continues for 23 GLT O-ring fixtures at 200 – 270 °F. Room temperature leak test failures have been experienced in all of the GLT O-ring fixtures aging at 350 °F and higher temperatures, and in 8 fixtures aging at 300 °F. The earliest 300 °F GLT O-ring fixture failure was observed at 34 months. The remaining GLT O-ring fixtures aging at 300 °F 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 °F for 96 - 120 months, or in GLT O-ring fixtures aging at 270 °F for 57 – 58 months. These aging temperatures bound O-ring temperatures anticipated during storage in K-Area Complex (KAC).

High temperature aging continues for 5 GLT-S O-ring fixtures at 200 – 300 °F. Room temperature leak test failures have been experienced in all of the GLT-S O-ring fixtures aging at 350 and 400 °F, and in one GLT-S O-ring fixture aging at 300 °F. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 - 250 °F for 78 months, or in the second GLT-S O-ring fixture aging at 300 °F for 78 months.

The GLT-S O-ring fixture aging at 300 °F which had just failed at the time the previous interim report was issued has since been opened and the O-rings examined. No unexpected conditions were observed.

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

Background

This is an interim status report for PCV O-ring experiments carried out per Task Technical Plan [1], which is part of the comprehensive 9975 package surveillance program [2].

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 [3] 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 °F, Viton® GLT O-rings aged at 270 – 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 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.

Twenty seven fixtures have been removed from the first test matrix for reasons other than failing the room temperature leak test. Further details of these fixtures are provided in Reference 4. Several fixtures conditioning at 300 °F have experienced room temperature O-ring leakage (i.e. failed), beginning in April 2010. The remaining 300 °F fixtures were retired in July 2012, still leak-tight after aging for more than 5 years. 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 than 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 2 E5 rad at a high dose rate (approximating a 10-year service dose at a bounding rate of 2 rad/hr). Gamma irradiation of fixtures in all test matrices is performed in a cobalt-60 cell [1].

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 was expected they would 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.

Several additional irradiations were performed in 2014, as proposed in Reference 5, as follows:

- Three of the 200 °F GLT fixtures received an additional 2 E5 rad (at high dose rate), and will continue to receive an additional 2 E5 rad every 4 years at the aging temperature.
- Three of the 200 °F GLT fixtures received an additional 1 E5 rad (at high dose rate), and will continue to receive an additional 1 E5 rad every 4 years at the aging temperature. These two steps will maintain an approximate balance relevant to storage conditions between the thermal and radiation degradation mechanisms.
- One retired, but unopened 300 °F GLT fixture was irradiated to total doses of 1 E6, 2 E6 and 3 E6 rad (bounding 50, 100 and 150 year doses). Each irradiation cycle was preceded and followed by a leak test, and also followed by a 24 hour cycle to 300 °F and another leak test. This provides a bounding radiation dose commensurate with the thermal degradation (5 years at 300 °F is comparable to about 155 years at the more realistic bounding temperature of 166 °F).
- Two un-used GLT O-rings were installed on a cone-seal plug and irradiated incrementally until significant damage was observed at 240 Mrad. The same was done for two GLT-S O-rings. The maximum dose rate received by the inner O-ring on the side oriented up in the irradiator was 3.5 E5 rad/hr. These irradiations provide a comparison between the two compounds in their radiation response.
- It was anticipated that the previous test would be followed by installing new O-rings in a fixture and repeating the irradiations, to identify whether the compression experienced within the O-ring groove would extend the useful service life under high radiation dose. This option was dropped after the O-rings failed to degrade in the manner expected.

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 1 E-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 2 E5 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 ~1.7 E5 rad/hr (lasting 72 minutes), as Viton[®] 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 1 E-7 ref-cc/sec air (2 E-7 cc/sec He) demonstrates leak-tightness when measured according to the requirements outlined in ANSI Standard N14.5-97 [3]. Initially, fixtures were also tested at their conditioning temperature. These additional tests were discontinued, as discussed in Reference 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 [3]. 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, subsequent testing has been 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 1, 2016.

A total of 23 GLT O-ring fixtures and 5 GLT-S O-ring fixtures remain in test. All of the GLT O-ring fixtures conditioning at 200 °F have remained leak-tight at room temperature, with total times at temperature ranging from 96 to 120 months (at the time of their last leak test). Two fixtures began conditioning at 270 °F in 2011. They have remained leak-tight at room temperature, with total time at temperature of 57 and 58 months (at the time of their last leak test). GLT O-rings in the remaining fixtures aging at 300 °F and higher were retired or failed previously, as noted in prior status reports. The times to failure for each GLT O-ring fixture are summarized in Table 3. Recent leak rate histories can be found in Table 4 for fixtures in test since the last status report [8].

Of the eight GLT fixtures conditioning at 300 °F that were retired from test in 2012, five have been maintained as-is for potential future test initiatives. One of these was used in 2015 for additional radiation exposure. In this test, fixture 51 was irradiated to a 50 year bounding dose (1 E6 rad total dose), and then heated to 300 °F for 24 hours. It was leak-tested initially, after irradiation and after heating. This process was repeated to 100 year and 150 year bounding doses. The fixture remained leak-tight throughout this process. Details are provided in Table 5.

Several GLT fixtures aging at 200 °F also received additional irradiation in 2014. Three fixtures (9, 41 and 42) received an additional 10 year bounding dose of 2 E5 rad, and will continue to receive this additional dose for every 4 years at temperature. Another three fixtures (53, 54 and 55) received half this additional dose (1 E5 rad), and will continue to receive this additional dose for every four years at temperature. These additional irradiations provide a continuing bounding dose (or a reduced, more realistic dose) that maintains an approximate balance with the thermal

aging, since four years at 200 °F is roughly equivalent to 10 years at the more typical O-ring temperature in storage of 166 °F. All six of these fixtures were leak tested before and after irradiation.

All of the GLT-S O-ring fixtures conditioning at 200 and 250 °F have remained leak-tight at room temperature, with total times at temperature of 78 months. One of the GLT-S O-ring fixtures conditioning at 300 °F was found not leak-tight after 69 months. Both of the O-rings in this fixture had failed. The second GLT-S O-ring fixture conditioning at 300 °F remains leak-tight after 78 months. GLT-S fixtures conditioned at 350 °F and at 400 °F failed as noted in previous status reports. The times to failure for each GLT-S 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].

The GLT-S O-ring fixture conditioning at 300 °F which failed (Fixture 34H) has been opened, and the O-rings examined. Upon opening the fixture, the O-rings showed no discernable springback (within 8 minutes - Figure 3). Subsequent dimensional measurements were used to calculate compression set. Twenty one minutes after opening, the compression set was 88 and 90% for the two O-rings. Two and four weeks later, compression set values showed almost no change, ranging from 86 to 90%. Values of compression set are calculated per ASTM D395 Method B and are summarized in Table 6. Additional observations from Fixture 34H include:

- The O-rings were adherent to the plug and the fixture body.
- A varying degree of brown / black stain was observed on the plug and fixture body, likely a combination of oxidized grease and material transferred from the O-rings. (Figure 4).
- A white residue, presumed to be oxidized grease, was observed on portions of the plug (Figure 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 °F, most of the GLT-S O-ring fixtures remained leak-tight longer than the GLT O-ring fixtures. At 350 °F, the trend is 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 °F, one GLT-S O-ring fixture has failed after 69 months, while multiple GLT O-ring fixtures had failed by this time. With this varying trend, it is premature to conclude from these data that 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.

No influence on time to failure has been observed from the irradiations performed on test fixtures. The additional irradiations added to the test matrix as the fixtures continue aging are similarly not expected to be severe enough to influence failure times. It is recognized that these irradiations present a simplistic approach that ignores potential synergistic effects from the simultaneous exposure to elevated temperature and radiation. The response to very high radiation doses noted in Reference 8 is similar between the two compounds, although GLT-S begins to soften at a lower dose than GLT. However, the doses involved are so much greater

than would be experienced in a reasonable storage scenario that this comparison would have no significant impact on storage service life.

A bounding steady state O-ring temperature of 199 °F has been calculated for an ambient temperature of 137 °F and 19 watts payload [9]. Reference 10 reports the maximum ambient temperature actually recorded (at that time) was 104 °F, although temperatures are more typically 90 °F or less. More recently, an average ambient temperature of 94 °F was recommended in Reference 11 for analysis of packages stored in KAC. This temperature is based on facility temperature measurements and provides margin in recognition of elevated temperatures within the storage array, temperature variation between the several storage areas, the potential addition of more packages in the future, and possible operational changes that might impact facility temperatures. With this bounding average ambient temperature of 94 °F, the maximum O-ring temperature is estimated to average 156 °F for a 19 watt package. In packages with lower internal heat loads, the O-ring temperatures would be correspondingly less.

The times at temperature for the fixtures to fail the room temperature leak test are shown graphically in Figure 6. In addition, the times at temperature for the fixtures still in test are also shown. A trend line provides a lower bound to the failure data to illustrate a potential extrapolation of the observed behavior to temperatures below 300 °F. In addition, the O-rings aging at 270 °F have now accumulated sufficient time at temperature to exceed the lower bound trend line, effectively demonstrating that the trend line is valid at least to that temperature. Note, however, that 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 °F might maintain a leak-tight seal for up to 75 years, and O-rings at KAC storage temperatures have the potential for even greater service life.

Conclusions

High temperature aging continues for 23 GLT O-ring fixtures at 200 – 270 °F. Room temperature leak test failures have been experienced in the GLT O-ring fixtures aging at 300 °F and above. No failures have yet been observed in GLT O-ring fixtures aging at 200 °F and 270 °F for 96 - 120 months and 57 – 58 months, respectively, which is more representative of (but still bounding to) O-ring temperatures during storage in KAC.

High temperature aging continues for 5 GLT-S O-ring fixtures at 200 – 300 °F. Room temperature leak test failures have been experienced in all of the GLT-S O-ring fixtures aging at 350 and 400 °F, and in one fixture aging at 300 °F. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 - 250 °F for 78 months.

The bounding average O-ring temperature expected in the KAC is approximately 156 °F, based on an average ambient temperature of 94 °F and the maximum payload (19W). Cooler ambient temperatures and reduced payloads are less challenging to the seals in storage. The data available to date suggest the GLT and GLT-S O-rings aging at 200 °F might maintain a leak-tight seal for up to 75 years, and O-rings at KAC storage temperatures have the potential for even greater service life. 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 KAC facility, the maximum ambient temperature is 137 °F [12], and the corresponding PCV O-ring temperature is 199 °F [9].
	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	2 E5 rad in 72 min	The bounding (high) dose rate for the PCV is 2 rad/hr. A total dose of 2 E5 rad represents ten years of storage (the initial period to be validated).
	Additional periodic 2 E5 rad at high dose rate	Additional dose after 8 years, and every 4 years subsequently, to maintain relative balance between degradation mechanisms (radiation, thermal)
	Additional periodic 1 E5 rad at high dose rate	Additional dose after 8 years, and every 4 years subsequently, to maintain relative balance between degradation mechanisms (radiation, thermal)
	2 E5 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 [13].
	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 [13].
	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	~2 E5 High	Normal	5, 6, 9, 27, 36, 37, 40			15, 16, 23, 24
200	~2 E5 High +2 E5 after 8 years	Normal	41, 42			
200	~2 E5 High +1 E5 after 8 years	Normal	53, 54, 55			
200	~2 E5 Low	Normal	10, 11			
200	No	Normal	1, 3, 43, 44, 56, 57			13, 28, 29
300	~2 E5 High	Normal		8, 12, 26, 31	7, 52	17, 22, 25, 39, 45, 46, 47, 58, 59, 60
300	~2 E5 High +2.8 E6 after retirement	Normal			51	
300	~2 E5 Low	Normal		32	18, 30*	21, 38
300	No	Normal		49, 33	4, 61	2, 14, 48, 50, 62
300	~2 E5 High	None				19
300	No	None				34
200	No	Krytox	35			
300	~2 E5 Low	Krytox			20	
GLT O-ring Fixtures – Second Test Matrix						
270	No	Normal	14W			
270	No	Normal	21W			
350	~2 E5 High	Normal		18D		
350	No	Normal		19D		
400	~2 E5 High	Normal		14D		
400	No	Normal		21D		
450	~2 E5 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	2 E5 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 (°F)	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			
34H	300	2013 - 2096	2013 - 2096
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 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/8/12	78	1.3 E-8
7/9/13	85	5.3 E-9
12/3/13	90	1.3 E-8
6/10/14	96	2.4 E-8
12/16/14	103	9.4 E-9
6/30/15	108	4.8 E-8
1/6/16	114	4.6 E-8

Test 3, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
5/14/13	84	5.9 E-9
11/12/13	90	5.8 E-9
5/13/14	96	1.3 E-8
11/11/14	102	2.9 E-9
5/19/15	108	1.2 E-8
11/17/15	114	7.9 E-9
6/1/16	120	5.5 E-8

Test 5, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/15/12	78	<1.6 E-9
7/25/13	84	8.7 E-9
1/22/14	90	2.7 E-8
7/29/14	96	7.9 E-9
2/3/15	102	1.1 E-8
7/29/15	108	2.8 E-8
2/10/16	114	1.5 E-8

Test 6, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/15/12	78	9.1 E-9
7/9/13	84	1.5 E-8
1/7/14	90	<2.5 E-9
7/8/14	96	3.8E-8
1/14/15	102	5.2 E-8
7/29/15	108	3.1 E-8
2/10/16	114	1.5 E-8

Test 9, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/9/13	72	<2.4 E-9
10/8/13	78	2.8 E-9
4/8/14	84	6.0 E-9
10/14/14	90	1.3 E-8
10/16/14	90	2.7 E-9 *
4/7/15	95	2.3 E-8
10/6/15	101	8.1 E-9
4/26/16	107	2.7 E-8

Test 10, 200 °F		
2E5rad /240 hr		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
9/10/12	72	1.6 E-8
5/14/13	78	<1.7 E-9
11/12/13	84	1.2 E-9
5/13/14	90	8.6 E-9
11/11/14	96	8.9 E-9
5/19/15	102	1.1 E-8
11/17/15	108	<2.3 E-9
6/1/16	114	3.2 E-8

* post-irradiation leak test

Test 11, 200 °F		
1.4E5 rad /479 hr		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/4/13	78	5.5 E-9
12/3/13	84	7.8 E-9
6-10-14	90	1.7 E-8
12/16/14	96	3.1 E-9
7/1/15	103	1.1 E-8
12/29/15	108	2.7 E-8
6/21/16	114	4.1 E-8

Test 27, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
12/11/12	61	5.8 E-9
8/13/13	67	8.2 E-9
1/22/14	72	5.4 E-9
7/29/14	78	1.6 E-8
2/3/15	84	1.1 E-8
7/30/15	90	2.2 E-9
2/10/16	96	108 E-8

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

Test 35, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/15/12	73	<1.6 E-9
7/9/13	78	1.7 E-8
1/7/14	84	9.8 E-8
7/8/14	90	3.8 E-8
1/14/15	96	1.2 E-7
6/30/15	102	2.8 E-8
1/6/16	108	4.6 E-9

Test 36, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/8/12	72	<1.9 E-9
6/4/13	78	<1.9 E-9
12/2/13	84	7.8 E-9
6/10/14	90	3.3 E-9
12/16/14	96	6.2 E-9
6/30/15	102	1.8 E-8
1/6/16	108	2.5 E-8

Test 37, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/8/12	72	2.7 E-8
6/4/13	78	<1.9 E-9
12/3/13	84	2.6 E-9
6/10/14	90	4.3 E-8
12/16/14	96	6.2 E-9
6/30/15	102	7.8 E-8
1-6-16	108	2.2 E-8

Test 40, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/9/13	72	2.2 E-9
10/8/13	78	2.8 E-9
4/8/14	84	1.8 E-8
10/14/14	90	7.7 E-9
4/7/15	96	5.7 E-8
10/6/15	101	1.3 E-8
4/26/16	108	2.0 E-8

Test 41, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/12/13	84	<2.9 E-9
5/13/14	90	3.9 E-9
11/11/14	96	1.5 E-8
11/17/14	96	5.8 E-9 *
5-19-15	102	1.8 E-8
12/29/15	109	5.5 E-9
6/21/16	114	1.9 E-8

* post-irradiation leak test

Test 43, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/8/12	72	2.7 E-9
6/4/13	78	<1.9 E-9
12/3/13	84	1.6 E-8
6/18/14	90	6.4 E-9
12/16/14	96	6.1 E-9
6/30/15	102	3.3 E-8
1/6/16	108	5.5 E-9

Test 42, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/13/13	84	1.7 E-8
5/13/14	90	2.9 E-8
11/11/14	96	8.9 E-9
11/13/14	96	1.4 E-8 *
5/19/15	102	2.7 E-8
11/17/15	108	1.3 E-8
6/1/16	114	2.5 E-8

* post-irradiation leak test

Test 44, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
5/14/13	78	<1.7 E-9
11/12/13	84	5.8E-9
5/13/14	90	1.2 E-8
11/11/14	96	3.0 E-9
5-19-15	102	1.8 E-8
11/17/15	108	9.0 E-9
6/1/16	114	1.8 E-8

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

Test 53, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/12/13	84	<2.0 E-9
5/13/14	90	5.5 E-8
11/11/14	96	2.1 E-8
11/13/14	96	7.2 E-9 *
5/19/15	102	1.2 E-8
11/17/15	108	9.0 E-9
6/1/16	114	1.4 E-8

* post-irradiation leak test

Test 55, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/12/13	84	2.9 E-9
5/13/14	90	4.0 E-8
11/11/14	95	1.2 E-8
11/17/14	95	5.8 E-9 *
5/19/15	102	1.5 E-8
12/29/15	109	2.4 E-8
6/21/16	114	2.2 E-8

* post-irradiation leak test

Test 57, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/8/12	72	<1.9 E-9
6/4/13	78	2.8 E-9
12/3/13	84	2.6 E-9
6/18/14	90	1.0 E-8
12/16/14	96	3.1 E-9
6/30/15	102	1.8 E-8
1/6/16	108	8.2 E-9

Test 21W, 270 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/24/11	6	<2.4 E-8
5/30/12	12	<3.0 E-8
8/13/13	25	5.5 E-9
7/8/14	35	1.6 E-8
7/24/15	48	4.7 E-8
2/10/16	54	4.2 E-8
6/1/16	58	2.5 E-8

Test 54, 200 °F		
2E5rad /72 min		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/12/13	84	<2.0 E-9
5/13/14	90	<2.7 E-9
11/11/14	96	2.4 E-8
11/17/14	96	5.8 E-9 *
5/19/15	102	3.6 E-8
11/17/15	108	1.4 E-8
6/1/16	114	1.8 E-8

* post-irradiation leak test

Test 56, 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
11/15/12	72	<1.6 E-9
7/9/13	78	1.1 E-8
1/7/14	84	7.4 E-8
7/8/14	90	1.6 E-8
1/14/15	96	6.6 E-8
7/29/15	102	1.6 E-8
2/10/16	108	9.1 E-9

Test 14W, 270 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
10/24/11	5	<2.6 E-8
5/30/12	12	<2.8 E-8
8/13/13	25	8.2 E-9
7/9/14	35	1.2 E-7
7/23/15	47	1.8 E-8
2/10/16	54	2.4 E-8
6/1/16	57	2.3 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 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
8/13/13	48	<1.9 E-9
2/20/14	54	1.6 E-8
9/4/14	60	3.4 E-8
3/3/15	66	1.4 E-8
9/9/15	72	3.0 E-8
3/15/16	78	2.7 E-8

Test 15H (GLT-S), 200 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
8/13/13	48	5.5 E-9
1/22/14	54	<1.9 E-9
7/29/14	60	5.9 E-9
2/3/15	66	<2.0 E-9
7/29/15	71	1.8 E-8
2/10/16	78	1.2 E-8

Test 16H (GLT-S), 250 °F		
No rad..		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
6/4/13	41	<1.9 E-9
12/3/13	48	7.8 E-9
6/10/14	54	3.3 E-9
12/16/14	60	6.1 E-9
6/30/15	66	2.6 E-8
1/6/16	72	8.2 E-9
6/21/16	78	2.5 E-8

Test 22H (GLT-S), 250 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/9/13	42	5.4 E-8
10/8/13	48	1.1 E-8
4/8/14	54	9.0 E-9
10/14/14	60	7.7 E-9
4/7/15	66	1.3 E-8
10/6/15	72	1.6 E-8
4/26/16	78	2.3 E-8

Test 29H (GLT-S), 300 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
4/9/13	42	9.9 E-9
7/9/13	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
10/14/14	60	1.2 E-6 *
1/14/15	63	1.7 E-7
4/28/15	66	1.7 E-8
7/29/15	69	2.6 E-8
10/27/15	71	3.4 E-8
2/18/16	75	1.7 E-8
5/18/16	78	8.6 E-8

* Apparent failure of 29H inner O-ring at 60 months. Both O-rings passed subsequent tests.

Test 34H (GLT-S), 300 °F		
No rad.		
Date	Time at Temp (months)	Leak Rate (std cc He/sec)
3/19/12	33	<2.2 E-8
7/11/12	36	<1.4 E-8
10/8/12	39	8.0 E-9
3/5/13	42	2.3 E-9
6/4/13	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
9-23-14	60	1.6 E-8
12/18/14	63	1.2 E-8
4/7/15	66	4.1 E-8
7/2/15	69	≥ E-5 *

* 34H both O-rings failed at 69 months.

Table 5. History of additional irradiations of fixture 51

Prior history: Retired July 2012 after 63 months at 300 °F. Verified leak-tight at that time. Fixture has remained unopened since then.		
Date	Action	Leak Rate (std cc He/sec)
9/10/14	Initial leak test	2.9 E-8
9/11/14	Irradiate to additional 8 E5 rad (1 E6 rad total)	1.3 E-8
9/24/14	Heat to 300 °F for 24 hrs	2.7 E-9
10/2/14	Irradiate to additional 1 E6 rad (2 E6 rad total)	1.1 E-8
10/8/14	Heat to 300 °F for 24 hrs	<2.0 E-9
10/13/14	Irradiate to additional 1 E6 rad (3 E6 rad total)	2.0 E-8
10/15/14	Heat to 300 °F for 24 hrs	<1.9 E-9

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 after failing room temperature leak test</i>						
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%
33 (1979 days at 300 °F)	10 minutes	88% / 82%	12 days	84% / 74%	29 days	84% / 73%
49 (1360 days at 300 °F)	14 minutes	84% / 81%	14 days	82% / 80%	30 days	81% / 79%
<i>GLT O-Ring Fixtures removed for other reasons</i>						
7 (2167 days at 300 °F)	19 minutes	93% / 84%	12 days	90% / 78%	29 days	90% / 75%
28 (630 days at 200 °F)	4 hours	68% / 62%	10 days	31% / 28%	230 days	28% / 24%
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%
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%
34H (2096 days at 300 °F)	21 minutes	90% / 88%	14 days	90% / 86%	28 days	88% / 89%
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.

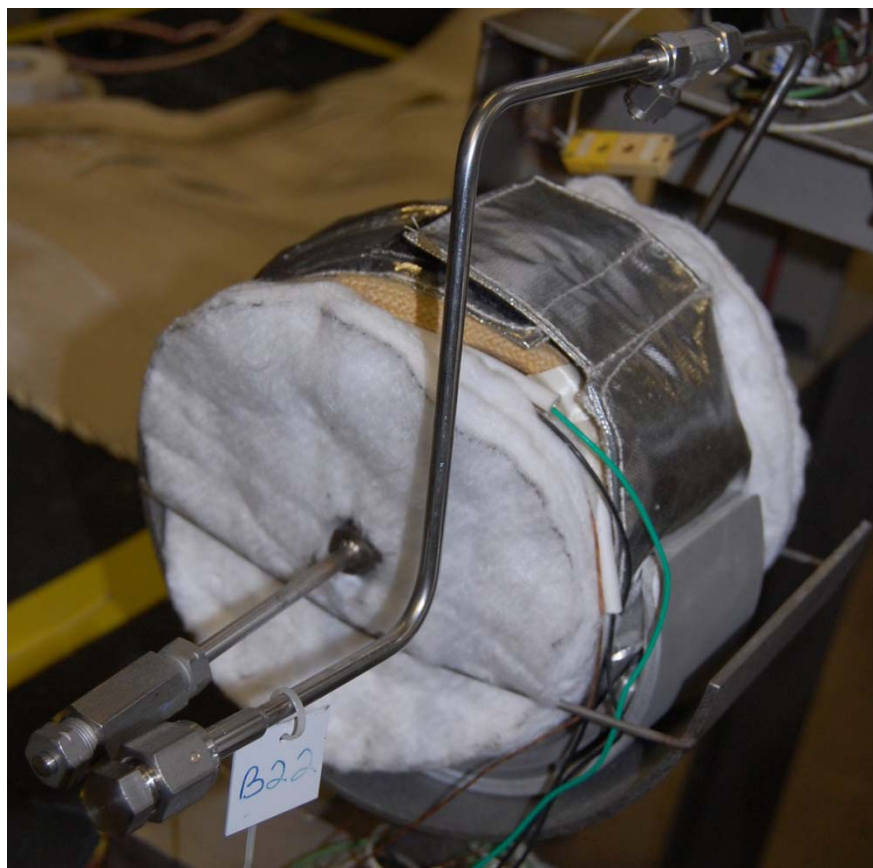


Figure 2. Assembled mock-up PCV.

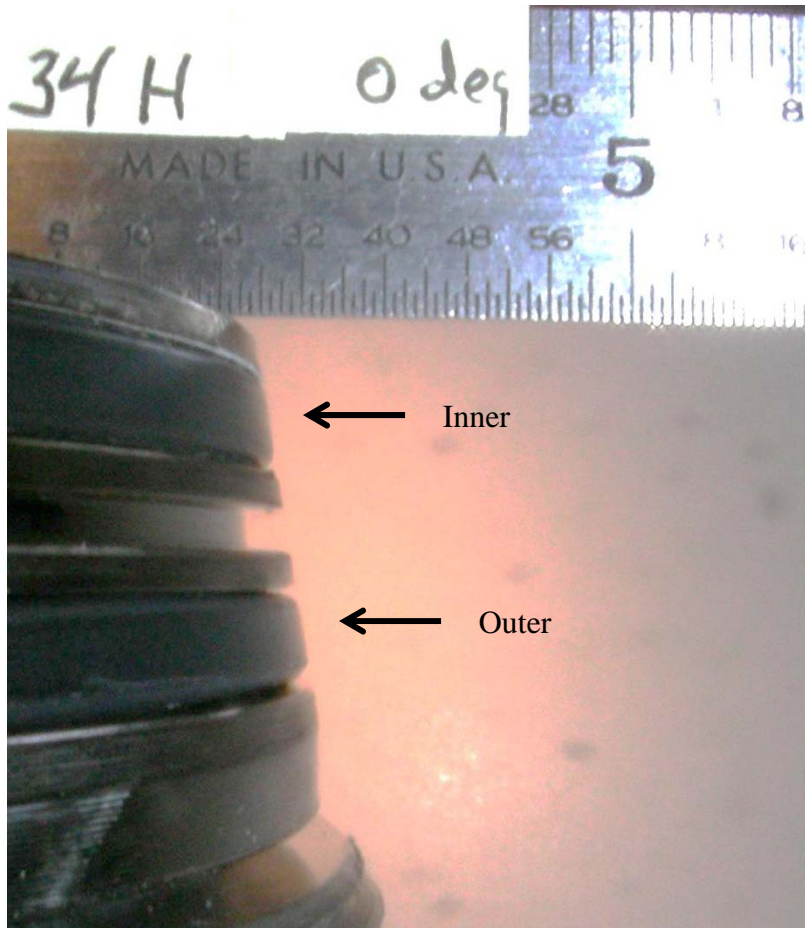


Figure 3. O-rings from fixture 34H prior to removal from the plug showing no discernible springback. Photograph was taken approximately 8 minutes after the fixture was opened.

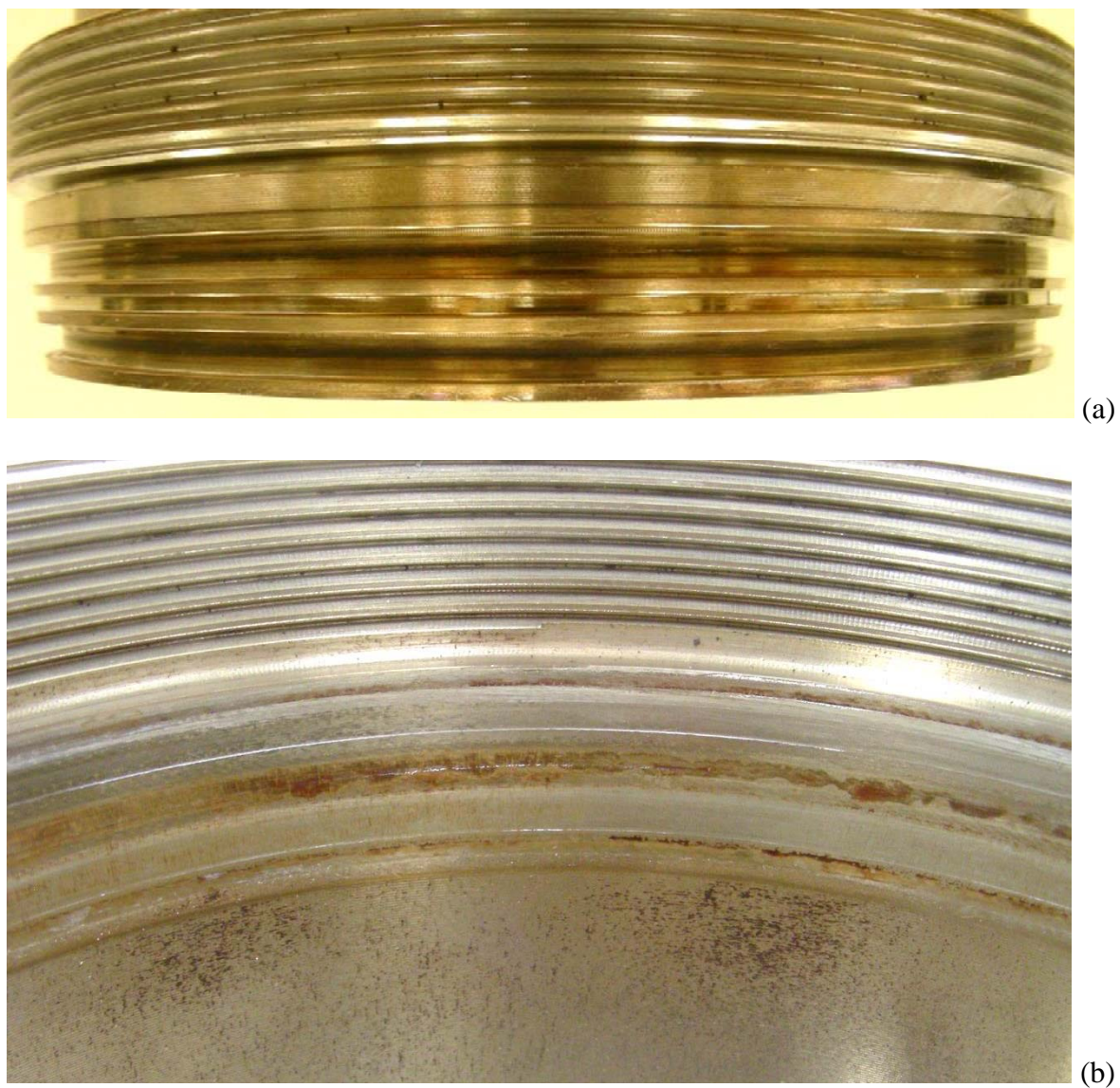


Figure 4. Stains on the plug (a) and body (b) from Fixture 34H.

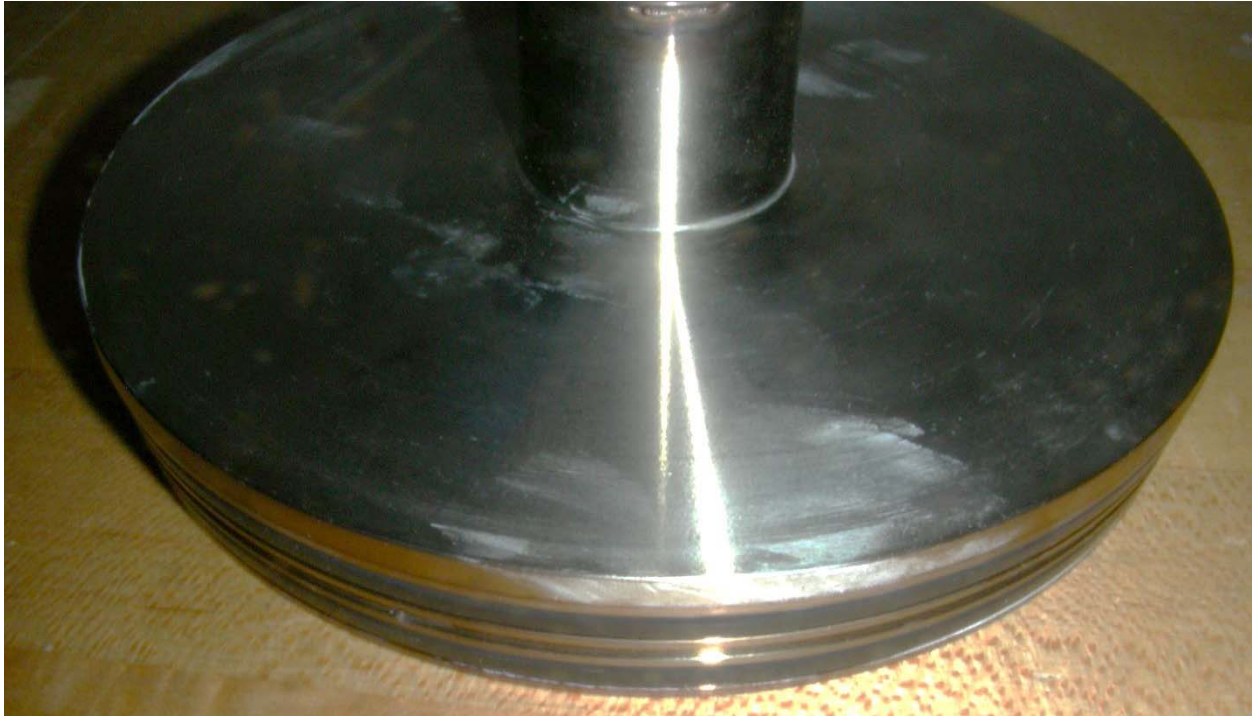


Figure 5. White residue observed on the plug from Fixture 34H.

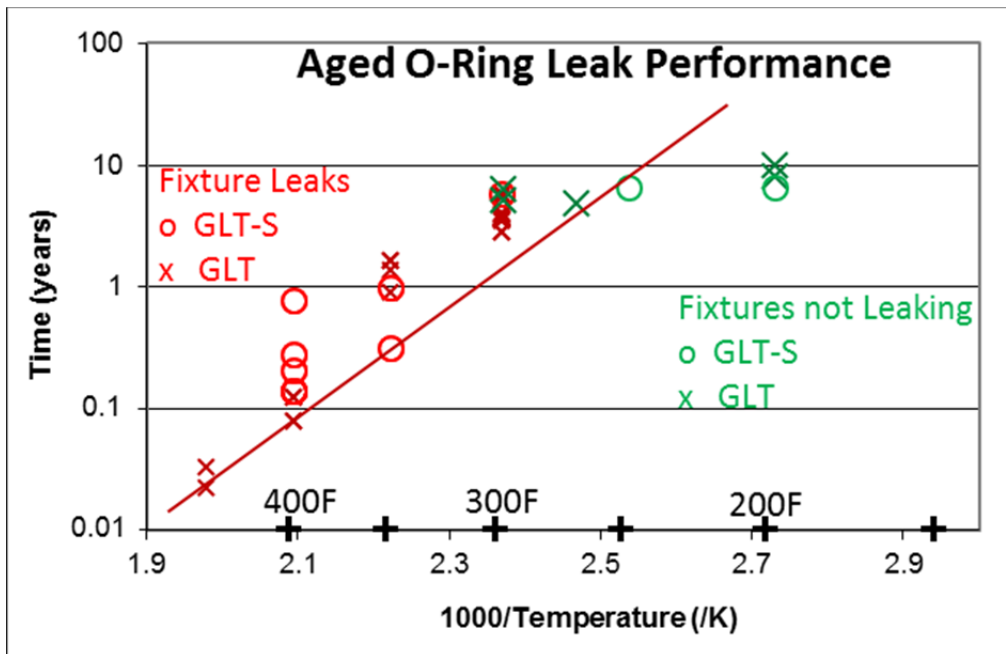


Figure 6. Summary of behavior for fixtures that failed the room temperature leak test and for fixtures still in test. The trend line illustrates a lower bound projection.

Distribution

G. A. Abramczyk, 730-A
J. S. Bellamy, 730-A
R. J. Bayer, 705-K
W. L. Daugherty, 773-A
K. A. Dunn, 773-41A
B. A. Eberhard, 105-K
B. L. Garcia-Diaz, 773-A
L. F. Gelder, 999-W
T. W. Griffin, 705-K
R. J. Grimm, 705-K
E. R. Hackney, 705-K
E. V. Henderson, 705-K
S. J. Hensel, 705-K
D. R. Leduc, 730-A
J. W. McEvoy, 707-C
A. J. McWilliams, 999-2W
W. E. Petty, 705-K
T. E. Skidmore, 730-A
D. E. Welliver, 705-K
K. E. Zeigler, 773-41A
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