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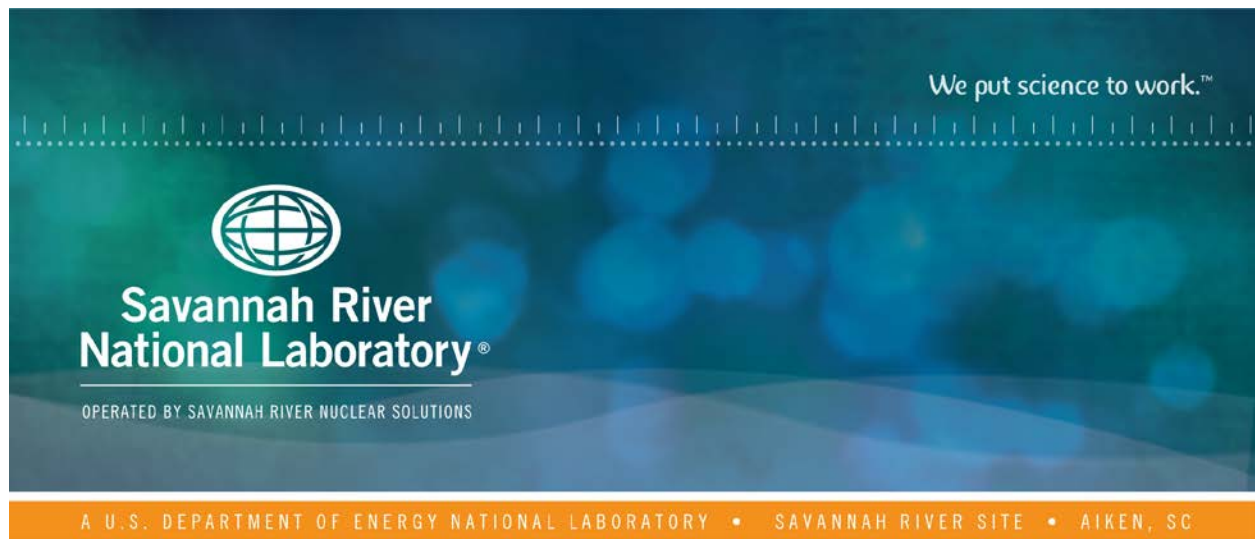
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# **FY2020 Status Report: Model 9975 O-Ring Fixture Long-Term Leak Performance**

**T. T. Truong**

August 2020

SRNL-STI-2020-00288, Revision 0



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T. T. Truong

August 2020

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Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.



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## EXECUTIVE SUMMARY

Leak testing experiments to monitor the aging performance of Viton® GLT and GLT-S O-rings used in the model 9975 shipping package has been ongoing since 2004 at Savannah River National Laboratory. Seventy tests using mock-up 9975 primary containment vessels (PCVs) with GLT O-rings were assembled and heated to temperatures ranging from 200 to 450 °F. In addition, fourteen tests with GLT-S O-rings were initiated in 2008 and heated to temperatures ranging from 200 to 400 °F. The conditioning temperatures are elevated compared to the calculated maximum O-ring temperature in a 9975 package in storage, 158 °F, to accelerate aging and enable observations of O-ring failures in a reasonable time frame.

The fixtures are leak tested periodically and all GLT O-ring fixtures aging at 350 °F or above have failed to maintain a leak-tight seal. Eight GLT O-ring fixtures aging at 300 °F have failed after 2.8 to 5.7 years at temperature while the remaining fixtures at 300 °F were retired from testing following more than five years without failure. No failures have yet been observed in GLT O-ring fixtures aging at 200 °F for 12 to 14 years or aging at 270 °F for 8.5 years. All GLT-S O-ring fixtures aging at 300 °F or above have failed their leak test. No failures have yet been observed in GLT-S O-ring fixtures aging at 200 and 250 °F for 10 to 10.5 years.

Data from the O-ring fixtures are generally consistent with results from compression stress-relaxation testing, and provide confidence in the predictive models based on those results. However, uncertainty exists in extrapolating these elevated temperature results to the lower temperatures of interest for normal storage in K-Area Complex (KAC). Oxygen consumption testing, which includes results from temperatures near KAC normal storage temperatures, is ongoing to provide further confidence and corroborate these extrapolations. The collective data from these test efforts suggest the minimum O-ring service life at KAC normal storage conditions should be at least 34 years for GLT and GLT-S O-rings.

Measurement of compression set in O-rings removed from failed fixtures, compared to that from KAC surveillance O-rings, indicates margin remains for O-rings still in service. Aging and periodic leak testing will continue for the remaining PCV fixtures.

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## **LIST OF ABBREVIATIONS**

CSR	Compression stress-relaxation
DE	Destructive examination
KAC	K-Area Complex
PCV	Primary containment vessel
SARP	Safety Analysis Report for Packaging
SCV	Secondary containment vessel
SRNL	Savannah River National Laboratory
SRS	Savannah River Site

## 1.0 Introduction

Special nuclear materials are stored within model 9975 shipping packages in the K-Area Complex (KAC) at the Savannah River Site (SRS). The 9975 shipping package consists of a 35 gallon stainless steel drum, Celotex® fiberboard insulation, lead shield, and primary and secondary containment vessels. The 9975 shipping package design, performance, and analysis for safe transport of radioactive material are described in the Safety Analysis Report for Packaging (SARP) [1]. The primary and secondary containment vessels (PCV and SCV) are sealed with dual Viton® GLT or GLT-S fluoroelastomer O-rings. The outer O-rings are credited with being leak-tight while in transport. The 3013 container is credited with providing containment of special nuclear materials while stored inside a 9975 package. For non-3013 containers stored in a 9975 package, the containment vessel outer O-rings are credited with maintaining containment [2]. The SRS Surveillance Program monitors material performance to establish a basis for service life and ensures the continued integrity of 9975 packages [3-4].

Experiments to monitor the aging performance of O-rings has been ongoing since 2004 at Savannah River National Laboratory (SRNL) and include leak testing, compression stress-relaxation (CSR) and oxygen consumption testing [5-6]. The experiments evaluate the performance of O-rings, conditioned at elevated temperatures, to predict their service life in 9975 packages stored in KAC. The performance of O-rings is evaluated by periodically leak testing mock-up PCV fixtures after conditioning at elevated temperatures. The 9975 shipping package is currently approved for a storage period of 20 years with 3013 and non-3013 containers in KAC [7-9].

Long-term testing of O-ring leak performance has been previously reported; reference 5 summarized experimental results of Viton® O-rings through June 2019. This report presents the cumulative data collected through June 2020.

## 2.0 Experimental Procedure

### 2.1 Fixture assembly and conditioning

Test fixtures were assembled with either Parker Seals V0835-75 (Viton® GLT) O-rings or Parker Seals VM835-75 (Viton® GLT-S) O-rings in a mock-up PCV. The assembly and setup have been described in detail in previous reports [10-11]. The two-piece lid of the mock-up PCV was machined to be identical to the actual PCV lid. The body of the mock-up PCV is 3.5 inches tall with a thread hole machined in the bottom to provide a port for evacuating and filling the vessel with gas. A PCV test fixture with the O-rings installed in the lid is shown in Figure 1a. The mock-up PCV fixtures were assembled per the requirements described in the SARP [1]. The fixture is heated with a flexible heater and capped with insulation at the exposed ends. The assembled fixture is shown in Figure 1b. The assembled fixture was leak tested initially to confirm leak-tightness before conditioning at 200, 250, 270, 300, 350, 400, or 450 °F.



**Figure 1.** Pictures of (a) mock-up PCV test fixture lid and body and (b) assembled fixture with heater and insulation.

## 2.2 Irradiation

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 ~240 hours) or a faster rate of ~1.7 E5 rad/hr (lasting 72 minutes), as Viton® and other polymers are known to be sensitive to dose rate. After irradiation, the fixture was leak tested again to ensure that irradiation alone did not affect leak-tightness, and then heated to conditioning temperature.

Several additional irradiations were performed in 2014 and 2018 to maintain an approximate balance relevant to storage conditions between the thermal and radiation degradation mechanisms. The fixtures were leak tested pre- and post-irradiation. Three fixtures received an additional 2 E5 rad (at high dose rate) and will continue to receive an additional 2 E5 rad every 4 years. Correspondingly, three other fixtures received an additional 1 E5 rad (at high dose rate) and will continue to receive an additional 1 E5 rad every 4 year. As of 2020, three fixtures have a total dose of 6 E5 rad and three fixtures have a total dose of 4 E5 rad.

## 2.3 Leak Testing

The O-ring fixtures are leak-tested after initial setup, after irradiation, and periodically after conditioning at elevated temperatures to the same leak-tight criterion as the 9975 PCV and SCV O-rings. The fixtures are leak tested per ANSI standard N14.5-97 at room temperature using a Varian 959 helium mass spectrometer leak detector [12]. Fixtures with leakage rates less than 1 E-7 ref·cc/sec air (or 2 E-7 ref·cc/sec He) are considered leak-tight. A gas filled envelope test, as defined in ANSI N14.5-97, 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. If a leak is found, it is possible to determine which O-ring is leaking by selectively directing the helium to either the fixture interior or exterior.

## 2.4 Test Matrix

Eighty-four fixtures have been assembled and evaluated since 2004 in three test matrices. The first test matrix was developed to determine the importance and effect of several variables on the condition of the GLT O-rings over time inside the KAC storage facility. The variables investigated include O-ring temperature, radiation/dose rate, and O-ring lubrication and are detailed in Table A-1. There are 62 fixtures and 22 separate sets of conditions in the first test matrix. Fixtures for the second test matrix were assembled using GLT O-rings in 2008 and aged at temperatures ranging from 350 to 450 °F. They were intended to provide some O-ring failures in a shorter time frame and to determine the time to failure at the vendor’s service temperature rating of 400 °F. The third test matrix investigated the leak performance of GLT-S O-rings using 14 fixtures and seven separate sets of conditions. 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. The status of these fixtures along with their test parameters is summarized in Table 1.

Experimental activities and analysis are documented in an electronic laboratory notebook [13].

**Table 1.** Summary of fixture status and test parameters

		Temperature (°F)	Gamma dose (rad), dose rate <sup>†</sup>	Lubricant	Fixture ID still in test	Fixture ID removed from test		
						Failed leak test	Retired July 2012	For other reasons**
<b>GLT O-rings</b>	1 <sup>st</sup> Test Matrix	200	~2 E5, High	Normal	5, 6, 27, 36, 37, 40			15, 16, 23, 24
		200	~2 E5, High +periodic 2 E5	Normal	9, 41, 42			
		200	~2 E5, High +periodic 1 E5	Normal	53, 54, 55			
		200	~2 E5, Low	Normal	10, 11			
		200	None	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, 45-47, 58-60
		300	~2 E5, High +2.8 E6 after retirement	Normal			51	
		300	~2 E5, Low	Normal		32	18, 30*	21, 38
		300	None	Normal		49, 33	4, 61	2, 14, 48, 50, 62
		300	~2 E5, High	None				19
		300	None	None				34
		200	None	Krytox <sup>®</sup>	35			
		300	~2 E5, Low	Krytox <sup>®</sup>			20	
	2 <sup>nd</sup> Test Matrix	270	None	Normal	14W, 21W			
		350	~2 E5, High	Normal		18D		
		350	None	Normal		19D		
		400	~2 E5, High	Normal		14D		
		400	None	Normal		21D		
		450	~2 E5, High	Normal		23D		
<b>GLT-S O-rings</b>	3 <sup>rd</sup> 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, 60H, 62H		
		400	2 E5, High	Normal		28H, 50H		

<sup>†</sup> Low dose rate: 667-830 rad/hr. High dose rate: ~1.7 E5 rad/hr

\* Has 1 failed O-ring (inner).

\*\* Other reasons fixtures were removed from test include high temperature leak test difficulties (11 fixtures), and unplanned temperature excursion (14 fixtures) [14].

### 3.0 Results and Discussion

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). A total of 23 GLT O-ring fixtures and 4 GLT O-ring fixtures remain in test. All GLT O-ring fixtures currently conditioning at 200 °F have remained leak-tight, with total times at temperature ranging from 12 to 14 years (at the time of their last leak test). Two fixtures began conditioning at 270 °F in 2011; they have remained leak-tight with total time at temperature of 8.5 years 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. All of the GLT-S O-ring fixtures conditioning at 200 and 250 °F have remained leak-tight, with total times at temperature of 10 and 10.5 years. GLT-S O-rings aging at 300 °F and higher have failed previously, as noted in prior status reports. The times to failure for each fixture are summarized in Table A-2; no fixtures have failed since the last status report. Leak rate histories can be found in Table 2 for fixtures in test since the last status report in 2019.

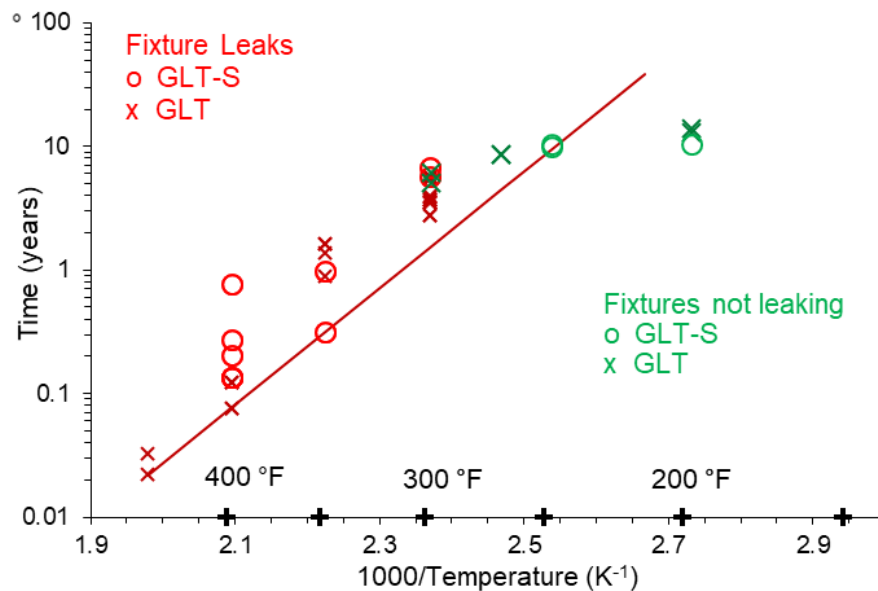
**Table 2.** Leak rate data since last status report

	Fixture ID	Temp. (°F)	Total $\gamma$ dose (rad), dose rate <sup>†</sup>	Date of leak test	Time at temp. (years)	Leak rate (std cc He/sec)	Date of leak test	Time at temp. (years)	Leak rate (std cc He/sec)
GLT O-rings	1	200	None	7/23/19	13	1.1 E-8	1/22/20	13.5	<2.5 E-9
	3	200	None	6/5/19	13	1.2 E-8	12/3/19	13.5	4.2 E-9
				6/2/20	14	7.4 E-9			
	5	200	2 E5, High	9/5/19	13	7.5 E-9	3/15/20	13.5	1.8 E-9
	6	200	2 E5, High	8/14/19	13	<2.5 E-9	2/12/20	13.5	3.4 E-9
	9	200	6 E5, High	11/12/19	12.5	4.9 E-9	6/2/20	13	7.4 E-9
	10	200	2 E5, Low	12/3/19	13	4.2 E-9	6/2/20	13.4	2.5 E-8
	11	200	1.4 E5, Low	7/16/19	12.5	9.6 E-9	1/15/20	13	<2.9 E-9
	27	200	2 E5, High	9/5/19	11.5	7.5 E-9	3/15/20	12	1.8 E-9
	35	200	None	8/14/19	12.5	1.4 E-8	2/12/20	13	1.4 E-8
	36	200	2 E5, High	7/16/19	12.5	1.6 E-8	1/15/20	13	8.5 E-9
	37	200	2 E5, High	7/23/19	12.5	1.1 E-8	1/22/20	13	<2.5 E-9
	40	200	2 E5, High	11/21/19	12.5	4.9 E-9	6/2/20	13	1.2 E-8
	41	200	6 E5, High	7/9/19	12.5	9.0 E-9	1/7/20	13	1.4 E-8
	42	200	6 E5, High	7/9/19	12.5	1.5 E-8	1/7/20	13	9.3 E-9
	43	200	None	7/16/19	12.5	6.4 E-9	1/15/20	13	8.5 E-9
	44	200	None	12/3/19	13	<3.0 E-9	6/2/20	13.4	9.8 E-9
	53	200	4 E5, High	12/3/19	12.9	4.2 E-9	6/2/20	13.4	2.0 E-8
	54	200	4 E5, High	7/9/19	12.5	1.2 E-8	1/7/20	13	1.4 E-8
	55	200	4 E5, High	7/9/19	12.5	1.2 E-8	1/7/20	13	1.4 E-8
GLT-S	56	200	None	9/5/19	12.5	3.7 E-9	3/15/20	13	<1.2 E-9
	57	200	None	7/23/19	12.5	1.1 E-8	1/22/20	13	<2.5 E-9
	14W	270	None	9/5/2019	7.8	1.1 E-8	12/3/19	8	4.2 E-9
				3/15/20	8.3	1.8 E-9	6/2/20	8.5	9.8 E-9
	21W	270	None	8/14/19	7.9	1.4 E-8	11/21/19	8.2	9.8 E-9
				2/12/20	8.4	5.8 E-8	6/2/20	8.7	7.4 E-9
	13H	200	None	10/9/19	10	4.5 E-9	4/21/20	10.5	7.9 E-9
	15H	200	None	9/5/19	10	3.7 E-9	3/15/20	10.5	<1.2 E-9
	16H	250	None	7/16/19	9.5	1.6 E-8	1/15/20	10	4.2 E-9
	22H	250	None	11/21/19	10	9.8 E-9	6/2/20	10.5	1.2 E-8

<sup>†</sup> Low dose rate: 667-830 rad/hr. High dose rate: ~1.7 E5 rad/hr

No influence on time to failure has been observed from the irradiations performed on test fixtures. Six fixtures received additional irradiation in 2018 and will continue to receive an additional dose every four years. As of 2020, three fixtures have a total dose of 6 E5 rad and three fixtures have a total dose of 4 E5 rad. It is recognized that these irradiations present a simplistic approach that ignores potential synergistic effects from the simultaneous exposure to elevated temperature and radiation.

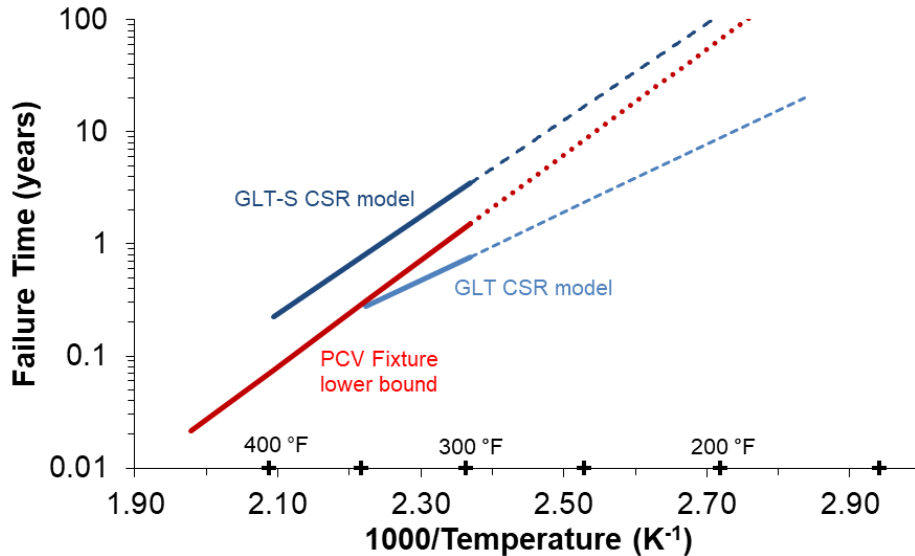
Recent thermal analysis of the 9975 package assumed 95 °F as the long-term average ambient temperature in KAC [15]. For a package with a maximum heat load of 19 W, the calculated O-ring temperature is 156 °F at beginning of life and 158 °F after 20 years of service with bounding package degradation. Figure 2 depicts the time at temperature for fixtures that failed the leak test and those that are still in test. A trendline provides a lower bound to the failure data to illustrate a potential extrapolation of the observed behavior to temperatures below 300 °F. O-rings aging at 270 °F have exceeded the lower bound trendline: they have been conditioning at temperature for over 8.5 years and still maintain a leak-tight seal. These O-rings demonstrate that the trendline maintains a lower bound to leak test data at least to 270 °F. Note, however, that the degree of scatter in the data would suggest significant uncertainty for extrapolations over a large temperature range. Nevertheless, this trendline suggests the possibility that O-rings aging at 200 °F might maintain a leak-tight seal for up to 74 years, and O-rings at KAC storage temperatures have a potential for even greater service life.



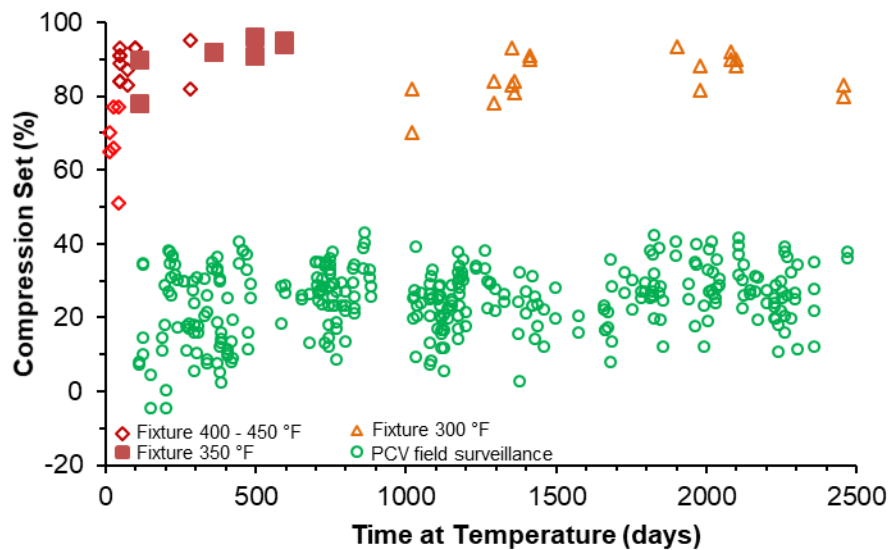
**Figure 2.** Summary of behavior for fixtures still in test and those that failed the leak test. The trendline illustrates a lower bound projection based upon failure data.

Figure 3 shows the lower bound trendline from leak testing results and predictions from compression stress-relaxation (CSR) data [6]. The dashed lines extrapolate the O-ring behavior through temperatures for which partial data exist (*i.e.* samples have not yet reached a failure point). Given the range of scatter observed in both fixture and CSR testing, it is recommended that any extrapolated service life estimates not exceed the minimum of these projections. CSR results indicate GLT and GLT-S O-rings will perform their required function in KAC storage conditions for at least 34 years. As additional data are collected, and the aging models refined, these service life estimates may be revised. However, at present, both GLT and GLT-S O-rings are expected to provide leak-tight seals well in excess of the current approved 20 year KAC service life for 9975 packages.

Compression set measures the amount of O-ring deformation after removal of force and was calculated using ASTM D395, Method B. A new O-ring should have a compression set of approximately 0% while a heavily degraded O-ring will have a compression set approaching 100%. The compression set of PCV O-rings in 9975 packages in KAC were measured previously and presented in Figure 4 along with the values from failed leak testing O-ring fixtures [10]. There is clear separation in compression set values for surveillance O-rings and failed fixture O-rings. While there are no data to specifically show how long it will take at storage conditions for the compression set to increase to that seen from failed test fixtures, as long as surveillance O-rings continue to show a similar behavior, they likely retain significant margin to reaching a failure condition.



**Figure 3.** Extrapolations of O-ring results for leak testing and compression stress-relaxation. The extrapolated segments shown by dashed lines are only partially supported by data which did not reach a failure condition.



**Figure 4.** Compression set values for field surveillance PCV O-rings and failed fixtures within one hour of opening vessel.

#### 4.0 Conclusions

Fixtures for GLT and GLT-S O-rings aging at 300 °F and above have experienced leak test failures or have been removed from test. No failures have yet been observed in GLT O-ring fixtures aging at 200 and 270 °F for 12 to 14 years and 8.5 years, respectively. For GLT-S O-rings, no failures have been observed for fixtures aging at 200 and 250 °F for up to 10.5 years. These 27 fixtures remain conditioning at elevated temperatures and will be periodically leak tested.

The PCV O-ring temperature expected in KAC is 156-158 °F based on an average ambient temperature of 95 °F and maximum payload of 19 W. The leak testing data to date suggest the GLT and GLT-S O-rings aging at 200 °F might maintain a leak-tight seal for up to 74 years, and O-rings in KAC storage at temperatures of 158 °F or less have the potential for even greater service life. Further consideration should be given to results from CSR testing which predict service life of up to 34 years. Measurement of compression set in O-rings removed during KAC surveillance, compared to failed fixtures, indicates margin remains for O-rings still in service in KAC. Both GLT and GLT-S O-rings are expected to provide leak-tight service well in excess of the current approved 20 year KAC storage life for 9975 packages.

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



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## Appendix A. Supplemental Information

**Table A-1.** Test matrix variables for O-ring fixtures

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 [16], and the corresponding PCV O-ring temperature is 199 °F [17].
	300 °F (149 °C)	The maximum allowable temperature for the PCV O-rings for continuous operation is 300 °F [18].
	270, 350, 400, 450 °F (132, 177, 204, 232 °C)	Elevated temperatures added to increase the likelihood of observing 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 >200 hr	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 [19].
	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 [19].
	None	It supplies comparative control data. Also, it is possible that the O-rings may be mistakenly installed without grease.

**Table A-2.** Summary of GLT and GLT-S O-ring leak failures

	Fixture ID	Temp. (°F)	Days at temperature to failure*	
			Inner O-ring	Outer O-ring
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	29H	300	2370 - 2455	2370 - 2455
	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 and the second time is the failed leak test. Failure occurred at some point between these two times.

**Distribution:**

G. A. Abramczyk, 730-A  
R. J. Bayer, 705-K  
J. S. Bellamy, 730-A  
A. J. Escobar, 705-K  
T. W. Griffin, 705-K  
R. J. Grimm, 705-K  
E. R. Hackney, 705-K  
S. J. Hensel, 705-K  
S. L. Hudlow, 705-K  
J. M. Jordan, 705-K  
M. D. Kranjc, 730-A  
D. R. Leduc, 730-A  
B. Lewczyk, 773-A  
J. W. McEvoy, 730-A  
M. J. Martinez-Rodriguez, 773-A  
L. Noll, 705-K  
R. A. Osborne, 705-K  
P. M. Palmer, 705-K  
M. M. Reigel, 773-A  
T. E. Skidmore, 730-A  
T.T. Truong, 773-41A  
K. E. Zeigler, 773-41A  
Document Control