

CSR Behavior and Aging Model for the Viton® Fluorelastomer O-Rings in the 9975 Shipping Package

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EXECUTIVE SUMMARY

The 9975 Type B shipping package is used within the DOE complex for shipping special nuclear materials. This package is re-certified annually in accordance with Safety Analysis Report for Packaging (SARP) requirements. The package is also used at the Savannah River Site as part of the long-term storage configuration of special nuclear materials. As such, the packages do not undergo annual recertification during storage, with uncertainty as to how long some of the package components will meet their functional requirements in the storage environment. The packages are currently approved for up to 15 years storage, and work continues to provide a technical basis to extend that period. This report describes efforts by the Savannah River National Laboratory (SRNL) to extend the service life estimate of Viton[®] GLT and GLT-S fluoroelastomer O-rings used in the 9975 shipping package.

O-rings of both GLT and GLT-S compositions are undergoing accelerated aging at elevated temperature, and are periodically tested for compression stress relaxation (CSR) behavior. The CSR behavior of O-rings was evaluated at temperatures from 175 to 400 °F. These collective data were used to develop predictive models for extrapolation of CSR behavior to relevant service temperatures (≤ 156 °F). The predictive model developed from the CSR data conservatively indicates a service life of approximately 37 years for Viton GLT O-rings at the maximum effective service temperature of 156 °F. The estimated service life for Viton GLT-S O-rings is significantly longer.

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LIST OF ABBREVIATIONS

CSR	Compression Stress Relaxation
KAC	K-Area Complex
PCV	Primary Containment Vessel
SCV	Secondary Containment Vessel
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TTS	Time-Temperature Superposition

1.0 Introduction

The Savannah River Site (SRS) stores packages containing plutonium (Pu) materials in the K-Area Complex (KAC). The 9975 shipping package is part of the approved storage configuration, and performs several safety functions, including impact resistance, containment, criticality prevention, and fire resistance to ensure the plutonium materials remain in a safe configuration during normal and accident conditions. The Model 9975 is a robust design for shipping, but it was not specifically designed for long term material storage. The 9975 surveillance program was established to identify potential degradation issues relating to long-term storage, and has focused primarily on the containment vessel O-ring seals and the fiberboard overpack material. The 9975 shipping package is currently approved for a storage period of 15 years, based on testing and analysis performed to date. The containment vessel O-ring seals are a specific focus of this assessment. This report describes efforts based on compression stress relaxation (CSR) testing to demonstrate that the O-ring seals have a service life beyond 15 years in this application. Leak testing of O-rings aging at bounding service temperatures is also being performed, with results reported separately.

The Model 9975 shipping package primary and secondary containment vessels (PCV, SCV) are each sealed with dual O-rings based on Viton[®] GLT (Parker Seals V0835-75) or GLT-S (Parker Seals VM835-75) fluoroelastomer. In early 2006, DuPont Performance Elastomers announced that the GLT polymer would no longer be produced in favor of the GLT-S polymer. Parker Seals subsequently qualified a new GLT-S compound (VM835-75) to the same Aerospace Material Specification AMS-R-83485 [1] that was initially written around the properties of V0835-75. DuPont literature and published papers [2] indicated that the new GLT-S polymer and test formulations are similar if not superior to the older GLT formulations in all respects. No negative behavior or lacking aspects were identified. In 2007-2008, SRNL/MS&T evaluated the new compound and determined suitability for shipping as well as for initial storage in KAC, as was previously done for the GLT O-rings. Chemical variation between the two base polymers is very minor (same monomers and monomer balance), with the primary difference being in the use of different cure site technology and end group efficiency.

Bounding O-ring service conditions include a maximum temperature of 94 °F average ambient temperature and 156 °F O-ring maximum temperature with a 19 W package. A recent evaluation of KAC ambient temperature data indicates a seasonal variation between 54 and 91 °F, with an overall average value of 74 °F [3]. This evaluation also demonstrated that the net degradation rate for this varying temperature profile was equivalent to the degradation rate for a constant temperature of 76 °F. Allowing for higher temperatures within an array of packages, increased numbers of packages stored and other future changes, Reference 3 recommended O-ring service life estimates be based on a constant ambient temperature of 94 °F. Previous analyses have assumed higher facility ambient temperatures and corresponding higher O-ring temperatures (up to 200 °F).

The O-rings are subjected to absorbed radiation dose rates of 2 rad/hr or less. Direct alpha exposure is not anticipated. Two O-rings are installed on a cone-seal plug in each containment vessel (Figure 1). The outer O-ring is credited as being leak-tight and provides the containment boundary. The inner O-ring provides a leak test volume and second containment barrier.

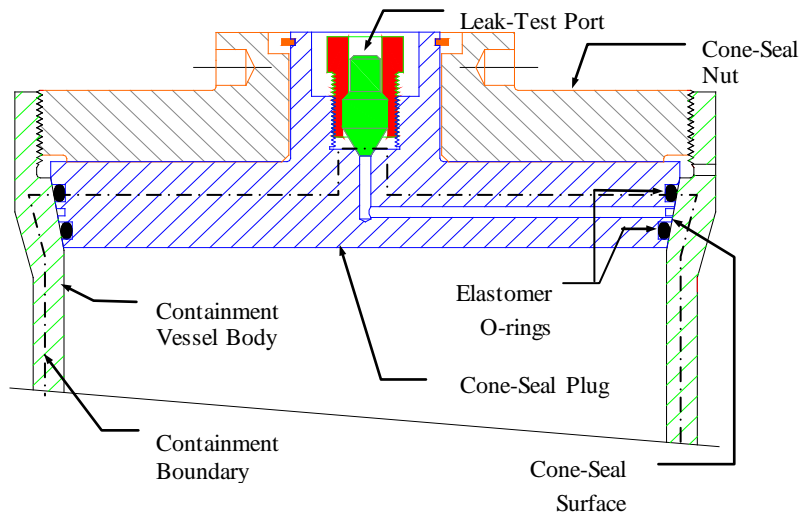


Figure 1: O-ring configuration in the 9975 containment vessels.

Laboratory-based accelerated aging testing uses CSR behavior as a primary tool for modelling O-ring degradation and predicting service life. In addition, leak testing of aged O-rings presented in SRNL-TR-2015-00205 [4] provides a more direct demonstration of a realistic leak-tight service life. Results from these tests can then be extrapolated to actual service temperatures based on appropriate mathematical models, which typically follow the form of an Arrhenius relationship.

Compression stress-relaxation (CSR) has become more widely accepted as a better measure of seal performance when compared to compression set behavior [5-9]. When an elastomer is compressed, the internal cross-linked polymer structure imposes a spring-like counterforce on mating surfaces to provide a seal. Over time, the sealing force is reduced due to physical and chemical relaxation processes. At some point, the seal may relax enough to allow unacceptable leakage. The relationship between sealing force and leakage is complex and very design specific. Leak rate depends on many variables including differential pressure, temperature, and design parameters such as surface finish, percent compression, gland fill, and lubricant use.

CSR is typically expressed as a force decay ratio (F/F_0) or as a percent of force retained. In studies on critical seals in nuclear weapon components, a sealing force ratio of $F/F_0 = 0.10$ (90% loss of counterforce) has been used to define the mechanical lifetime of an elastomer [5-7]. Though somewhat arbitrary, this value provides some residual counterforce to accommodate reasonable thermal or mechanical changes. For a highly static seal, practically all sealing force may be lost before leakage occurs, although it may not respond favorably to changes in the service environment.

O-ring performance is defined by leak-tightness, per ANSI standard N14.5-97 [10] at room temperature. Leak-tightness of the O-rings is related to the retention of mechanical properties. As long as the polymer retains a certain degree of counterforce against the sealing surfaces when compressed, a leak-tight seal is maintained. The minimum counterforce required to maintain leak-tightness in the 9975 seal design has not been established, but is believed to be quite low.

Some investigators have suggested threshold force values of 1 N/cm, though not as an absolute value for all designs [5].

2.0 Experimental Procedure

CSR behavior is being measured per ASTM D6147 using Shawbury-Wallace C11 jigs and a Wallace Mark IV relaxometer. This ASTM standard allows for either constant or periodic measurement of sealing force. The current work employs the periodic measurement approach, which is conducive to longer test periods and more realistic aging temperatures. Size 2-213 O-rings were used to fit within the CSR jigs, while matching the thickness of the 9975 O-rings (0.139 inch). To mimic the 9975 O-ring design, the O-rings are tested with a nominal 18% compression imposed during aging. In addition, some test O-rings also incorporate the 20% nominal ID stretch from the containment vessel design. The use of inserts to obtain this ID stretch also mimics another aspect of the seal design – the O-ring is further protected from constant exposure to fresh air. This degree of ID stretch is notably higher than the <5% typically recommended by seal manufacturers, but so far has not affected seal performance.

The aging temperatures selected for the CSR tests were: 175, 235, 250, 300, 350 °F (both compounds) and 400 °F (GLT-S only). The 175 °F value approximated the peak O-ring temperature anticipated for 104 °F ambient (the maximum ambient temperature identified in KAC) based on calculations at the time aging began. The 300°F value is the vessel design limit. The 350 °F temperature was selected to challenge the O-rings in a shorter aging period but is less than the “continuous” O-ring service rating of 400 °F. The other aging temperatures provide intermediate points of comparison. Some O-rings were gamma-irradiated to a 50-year dose of 0.88 Mrad at a rate of 0.44 Mrad/hr prior to thermal aging. The aged O-rings are periodically removed from their aging environment to measure the remaining counterforce, with an assumed end-of-life criterion of $F/F_o = 0.10$. The CSR methodology has been previously described in greater detail [11].

For reference, Table 1 provides equivalent temperature values used in this report.

Table 1: Equivalent temperature values.

°F	K	1000/K
150	339	2.95
175	352	2.84
235	386	2.59
250	394	2.54
300	422	2.37
350	450	2.22
400	477	2.10

3.0 Results and Discussion

The reported compression-stress-relaxation values are the average of five sequentially measured counterforce values for each aging period, minus the empty jig break force, normalized to the

initial maximum value. The measured counterforce values tend to decrease slightly during a given measurement cycle due to jig cooling. The CSR values obtained over aging periods of up to 7 years for the GLT and GLT-S O-rings are shown in Figure 2 and Figure 3, respectively. As expected, the higher the aging temperature the more rapid the counterforce decay. The overall trend indicates a quicker decay of the GLT than the GLT-S at a given temperature, consistent with published references.

A comparison of pretreatments (with and without stretch or irradiation) is shown in Figure 4 for an aging temperature of 250 °F for GLT-S O-rings. Although some small range of CSR behavior is shown, the spread of pretreated O-ring behavior lies primarily within the range of the non-pretreated O-ring behavior, implying that physical and radiological aging factors will have a minimal effect on the CSR performance at the conditions anticipated. This trend is similar for both sets of GLT and GLT-S O-rings. Tests at 250 °F were ceased before reaching a failure condition primarily for this reason.

The entire CSR data sets for the GLT and GLT-S O-rings are given in Figure 5 and Figure 6, respectively. For clarity, all CSR data are shown, plotted separately by temperature, in Appendix A. The data sets are not entirely complete due to external factors such as power outages and equipment malfunctions. These are described further in Appendix A.

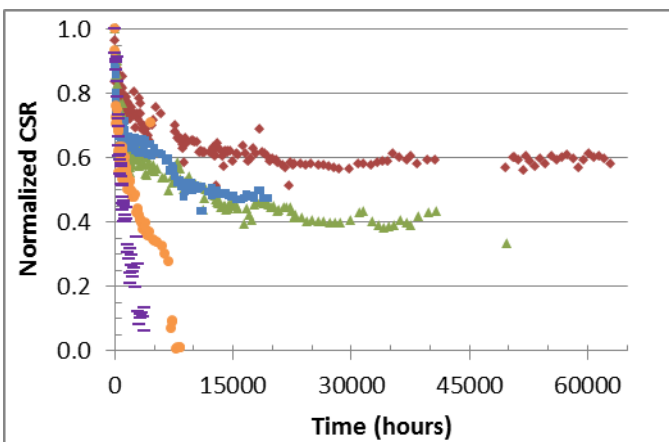


Figure 2: Representative normalized CSR values for GLT O-rings aged at:

◆ 175 °F, ▲ 235 °F, ■ 250 °F,
● 300 °F, and ✖ 350 °F.

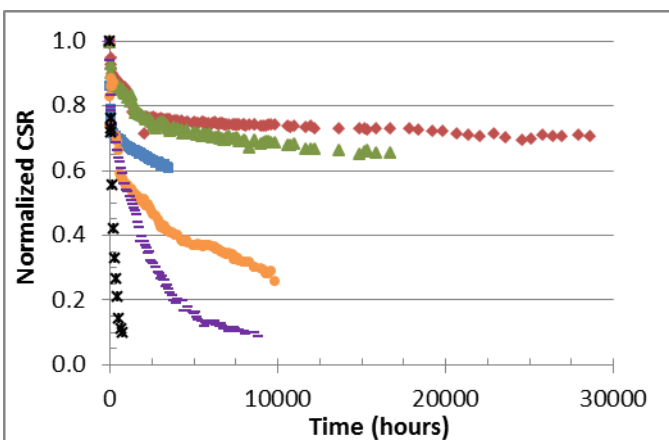


Figure 3: Representative normalized CSR values for GLT-S O-rings aged at:

◆ 175 °F, ▲ 235 °F, ■ 250 °F,
● 300 °F, ✖ 350 °F, and * 400 °F.

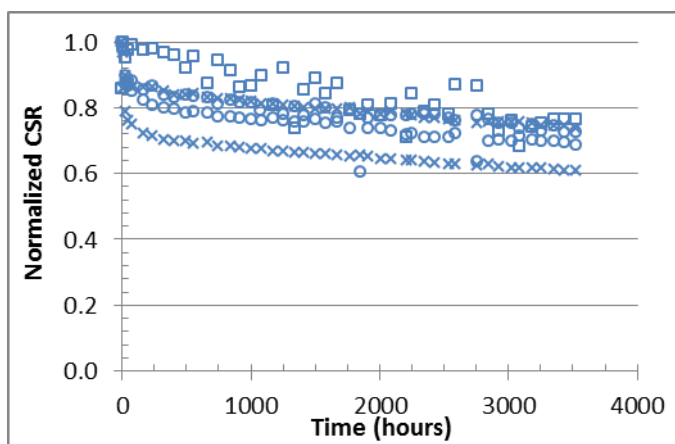


Figure 4: All GLT-S O-rings aging at 250 °F including O-rings that have been stretched and irradiated prior to aging:

× Baseline,
□ Stretched
○ Irradiated.

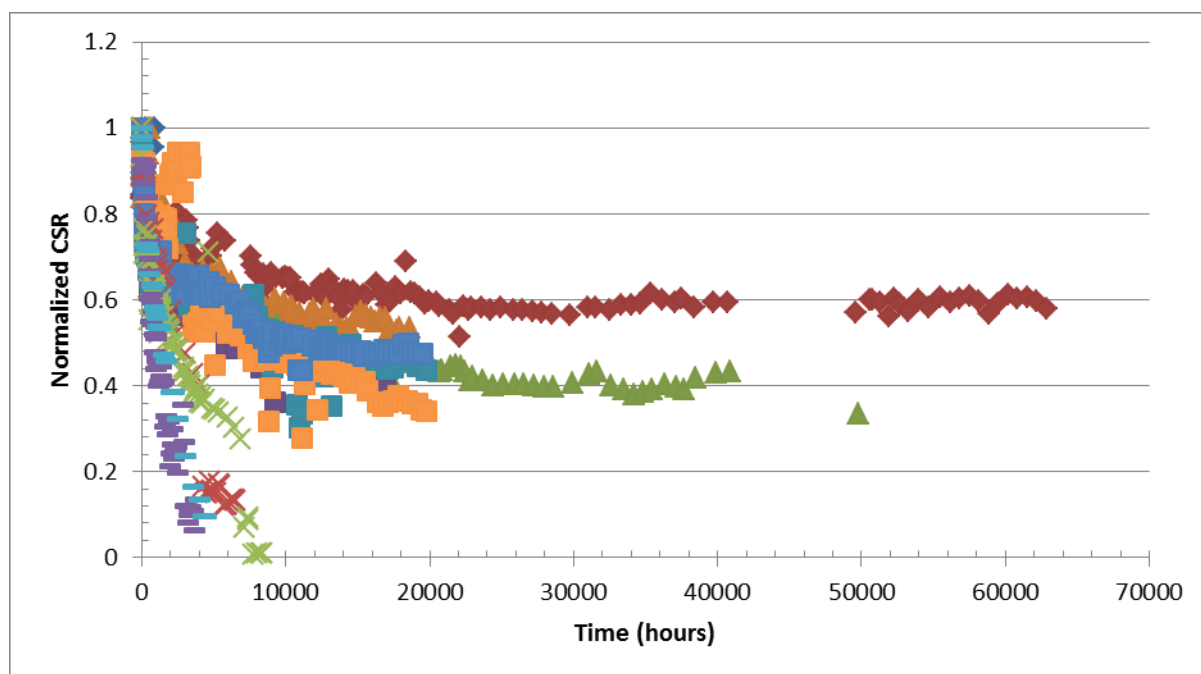


Figure 5: CSR values for all tested GLT O-Rings:

◆ 175F J-1B	◆ 175F J-8 Stretched/Irradiated	▲ 235F J-2B	▲ 235F J-9
■ 250F J-3B	■ 250F J-10	■ 250F J-16	■ 250F J-17
× 300F J-4B	× 300F J-18	■ 350F J-21	■ 350F J-7B

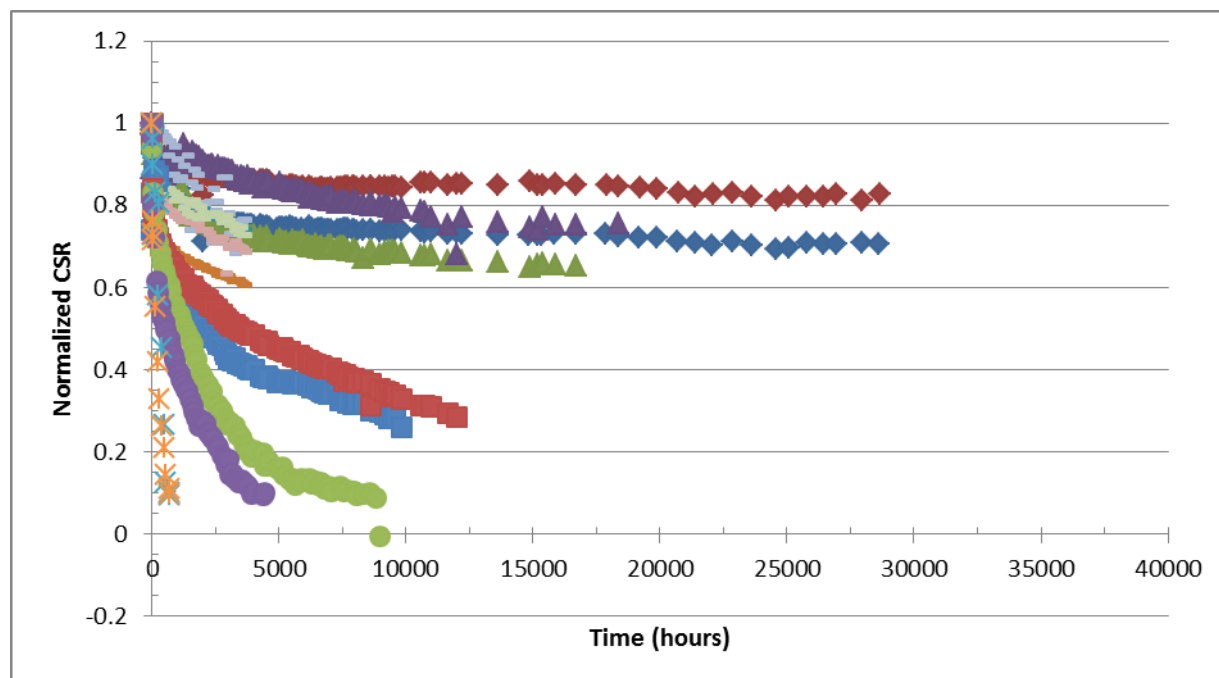


Figure 6: CSR values for all tested GLT-S O-rings:

◆ 175F J-51 ◆ 175F J-52 ▲ 235F J-53 ▲ 235F J-54 — 250F J-55
 — 250F J-56 — 250F J-57 Stretched — 250F J-58 Irradiated — 250F J-59 Irradiated — 300F J-60
 ■ 300F J-61 ● 350F J-62 ● 350F J-63 * 400F J-6B * 400F J-25

Discussion

Standard time-temperature superposition (TTS) [8] is applied to the CSR data for GLT and GLT-S O-rings, Figure 7 and Figure 8, with both data sets shifted to a reference temperature of 175 °F. These show reasonable agreement in overall curve shape for each O-ring type, providing an indication that a common degradation mechanism(s) is active over this temperature range. The only departure from this trend is for the highest temperature for each material (350 °F for GLT and 400 °F for GLT-S). At these extremes, the shifted data tend to be a little steeper than the other time-shifted curves, and an alternate degradation mechanism may be contributing somewhat to the overall behavior. In absence of significant oxidation or chemical changes, much of the observed relaxation is presumed to be mechanical in nature. The TTS curves for the two materials are given in Figure 9. The individual curves are all the TTS curves for a given O-ring type that were compiled into a single curve using a 9 point average and are intended as a qualitative visual representation. It can be seen from this comparison that the time required for the counterforce to drop to 0.1 F_0 is about 10 times longer for GLT-S than for GLT. The shift factors, a_T , are plotted in Figure 10 for the two materials. The vertical spread of data points at a given temperature is due to selecting a unique shift factor for each sample at that temperature, rather than selecting an average shift factor.

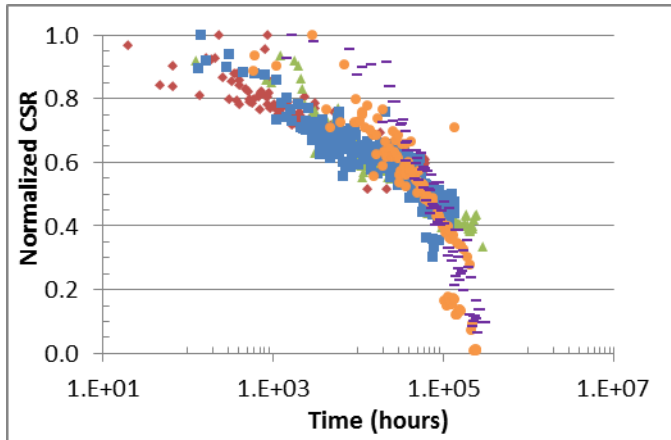


Figure 7: Time-temperature superposition of GLT O-ring data at 175 °F aged at:

◆ 175 °F, ▲ 235 °F, ■ 250 °F,
● 300 °F, and ■ 350 °F.

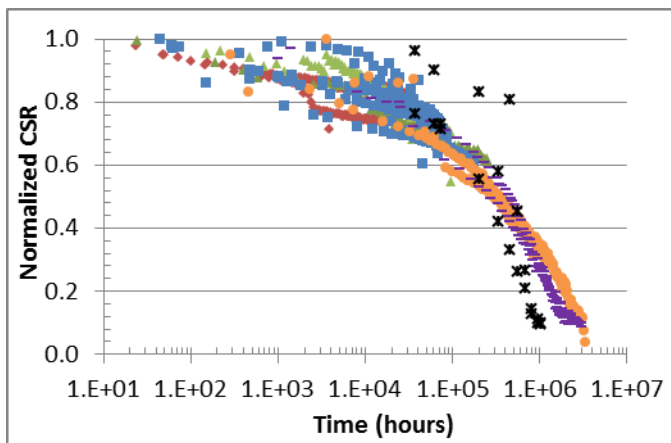


Figure 8: Time-temperature superposition for GLT-S O-ring data at 175 °F aged at:

◆ 175 °F, ▲ 235 °F, ■ 250 °F,
● 300 °F, ■ 350 °F, and * 400 °F.

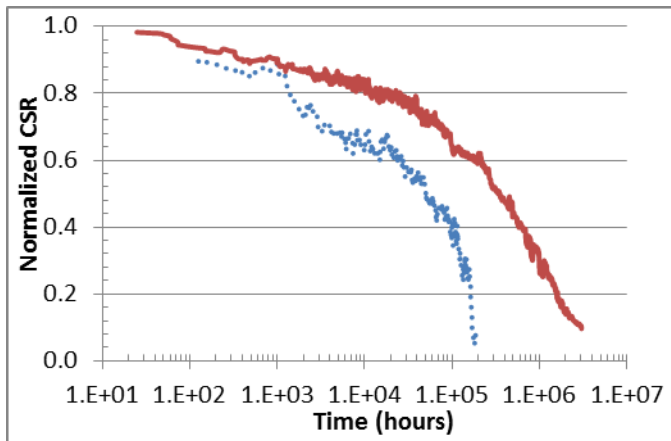


Figure 9: Comparison of TTS curves for both GLT (.....) and GLT-S (—) O-rings using a 9 point average of all available data.

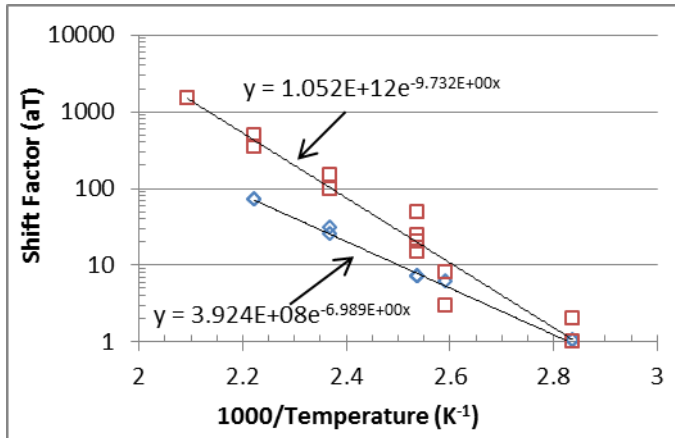


Figure 10: Both (♦) GLT and (■) GLT-S shift factors (a_T) plotted for comparison.

When the shift factors are plotted on a semi-logarithmic plot as a function of inverse temperature, results that form a straight line are indicative of Arrhenius behavior, described by

$$\text{degradation rate} = Ae^{-E_a/RT}$$

The slopes provided by the shift factors in Figure 11 can be used to calculate the activation energy ($= -\text{slope} * \text{ideal gas constant}$). This gives activation energy estimates of 60 kJ/mol for GLT and 81 kJ/mol for GLT-S. With the activation energy, the time to reach 0.1 F_0 can be extrapolated for any service temperature of interest, provided the active degradation mechanisms remain constant over the temperature range. For example, the TTS curve for GLT at 175 °F is shown in Figure 11 with a further time shift to show the expected behavior at the highest expected service temperature of 156 °F. This shift indicates an approximate 1.9x increase in the time required to reach a 0.1 F_0 CSR value (approximately 37 years at 156 °F). Figure 12 shows a similar extrapolation for the GLT-S O-ring series with a 2.3x increase in the time required to reach a 0.1 F_0 CSR value (approximately 791 years at 156 °F). Extrapolations to additional temperatures are provided in Table 2 and Table 3 for GLT and GLT-S O-rings, respectively. Since this extrapolation is only based on this set of CSR data, it does not include degradation mechanisms or reactions that are not present under the existing testing parameters, such as surface modifications, microcracking, stretching or other effects that could lead to an O-ring leak.

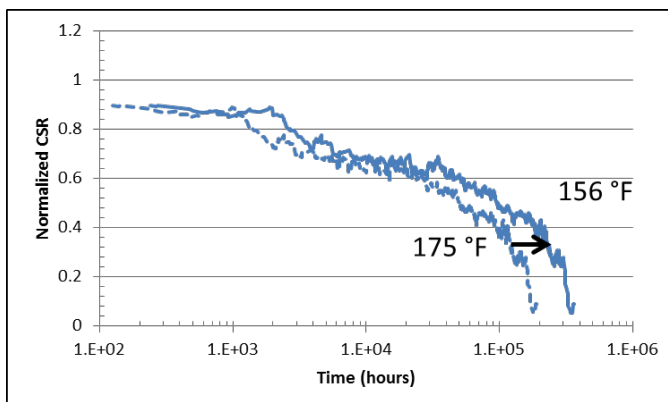


Figure 11: Extrapolation of the TTS GLT 9 pt averaged curve at 175 °F (.....) to 156 °F (—).

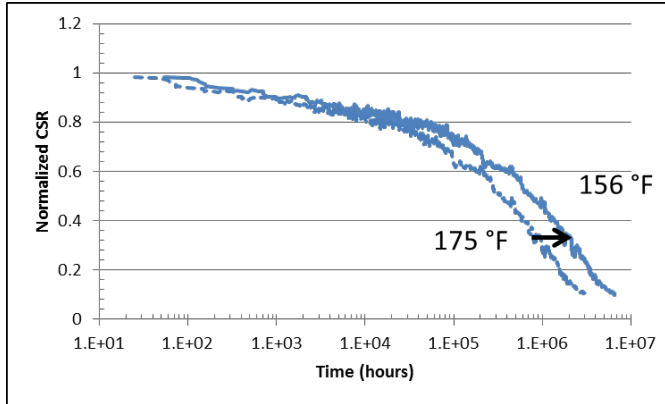


Figure 12: Extrapolation of the TTS GLT-S 9 pt averaged curve at 175 °F (.....) to 156 °F (—).

Table 2: Extrapolated lifetimes for GLT O-rings based on the time to reach CSR=0.1

T (°F)	t(csr=0.1) (hr)	t (yr)
175	170000	19
156	323000	37
150	396000	45
125	958000	109
100	2510000	286

Table 3: Extrapolated lifetimes for GLT-S O-rings based on the time to reach CSR=0.1

T (°F)	t(csr=0.1) (hr)	t (yr)
175	3014000	344
156	6932000	791
150	9077000	1036
125	56560000	3544
100	1.19E+08	13528

There is no demonstrated correlation between CSR behavior and leak-tightness for this O-ring configuration, although it is likely that both behaviors are strongly related to the same degradation mechanisms, i.e. as the O-ring counter force drops, the ability to maintain a seal against transient conditions will also decrease. Sandia National Laboratory personnel have suggested a minimum threshold force value of 1 N/cm to maintain leak-tightness for a seal design they were studying [5]. While not directly applicable to the 9975 O-rings, this value provides a convenient comparison point. The O-rings used in the current work have an average diameter of 1.36 inches (for a total length of 3.45 cm). These O-rings exhibited an initial counterforce at aging temperature of ~580N up to > 1000N. With the assumed failure criterion of 0.1 F_0 , these O-rings retain a contact force of ~17 N/cm or greater at end of life. Compared to the Sandia threshold, this would suggest that the CSR failure criterion is probably conservative to the actual leak-tight lifetime.

Reference [4] summarizes leak behavior and develops a service life estimate based on time to leak failure of approximately 75 years at a temperature of 200 °F. For this estimate both GLT and GLT-S data are treated as a single population due to significant scatter and overlap in data. With leak test failures only at the higher test temperatures (300 °F and above) to date and the scatter in the data, this extrapolation includes significant uncertainty. However, this estimate is roughly consistent with the CSR data for GLT-S O-rings.

It should be noted that there are currently no aging data at temperatures below 175 °F. It is assumed that the observed behavior at higher temperatures can be extrapolated to lower service temperature based on the Arrhenius relationship. However, it is possible that the active degradation mechanisms could change at lower temperatures, and such extrapolations would be invalid. Until data become available relevant to lower temperatures, the extrapolations presented in this report should be used with caution.

4.0 Conclusions

From the CSR data, the stress-relaxation behavior of O-rings based on Viton[®] GLT-S is superior to that of O-rings based on Viton[®] GLT, consistent with the limited literature data. The sealing force decay is slower for GLT-S O-rings, and the estimated service life at operating temperatures is greater.

The data provide confidence that significant O-ring service life exists at actual service temperatures to support an extension in the approved storage period. With varying degrees of conservatism, the following service life estimates can be made:

- For a reference temperature of 175 °F, the analysis indicates a service life of approximately 20 years for Viton GLT O-rings in the 9975 package, and approximately 340 years for Viton GLT-S O-rings.
- At a more realistic but bounding O-ring temperature of 156 °F (based on actual KAC ambient temperature measurements) the estimated service life is 37 and 791 years, for GLT and GLT-S, respectively.
- Longer service lives are possible given that the majority of packages in storage contain significantly less than the maximum 19W payload, with lower internal temperatures.

Since there are no aging data currently available at temperatures below 175 °F, the CSR aging behavior at 175 °F is quite stable, and the possibility exists for changes in the active degradation mechanisms at lower temperatures, these extrapolated service life estimates should be used with caution until such data become available.

5.0 Recommendations, Path Forward or Future Work

Additional work could be designed to give a higher degree of confidence to the data and focus on other factors contributing to the degradation of the sealing nature of the O-rings such as additional samples at existing conditions or intermediate temperatures. However, the time to reach meaningful sealing force decay at lower, more realistic temperatures would be extensive.

Also thermal cycling, calculations based on more realistic service parameters, and alternative inert environments would increase the similarity of testing parameters to actual service conditions. Testing to correlate leak performance and sealing force decay could also be performed, though current data suggests that sealing force decay well below 0.1Fo is required for leakage. CSR testing at 175°F will continue to provide a leading indicator of performance at bounding storage temperatures.

6.0 References

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Appendix A

The entire data sets are presented for the CSR values of the GLT and GLT-S O-rings tested at each temperature. The O-rings with additional pretreatment are noted in the respective captions.

CSR Results for GLT Samples:

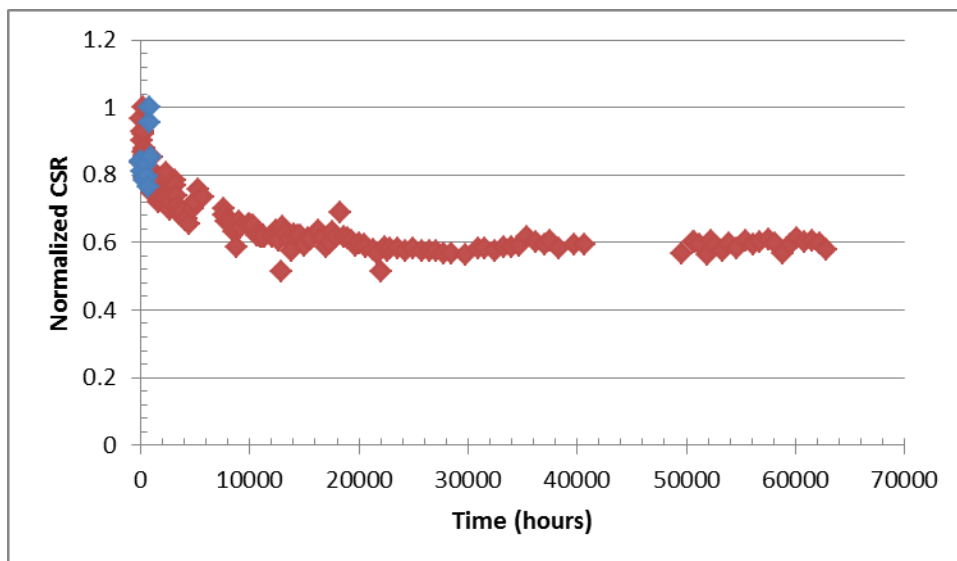


Figure A1: CSR values for GLT O-rings aged at 175 °F for no pretreatment, ◆, and stretched and irradiated pretreatment, ◆, showing similarity over the limited range of values for both pretreatments. The stretched and irradiated sample remains in test. No data were recorded between ~42000 and 50000 hours.

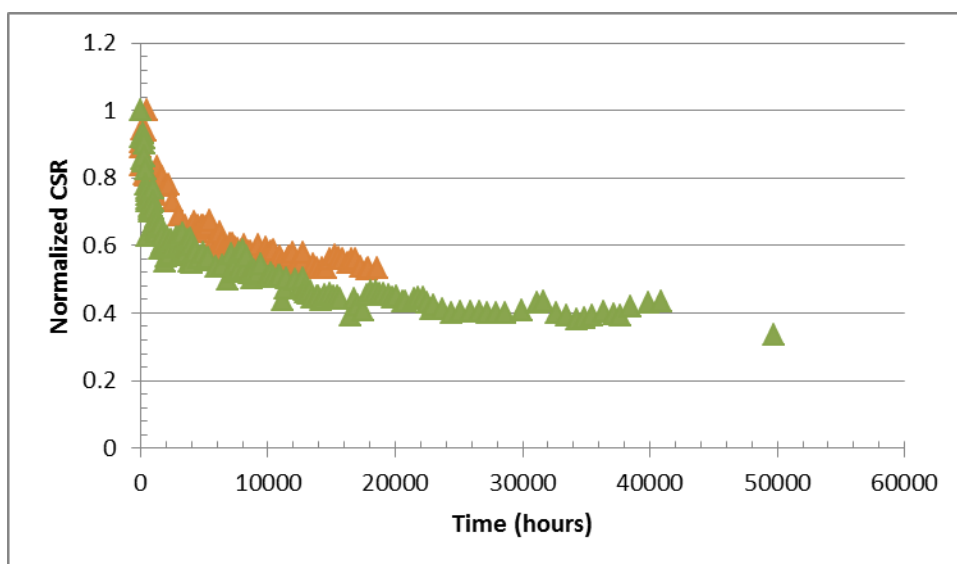


Figure A2: CSR values for GLT O-rings aged at 235 °F. Samples were terminated following a temperature excursion in April 2013.

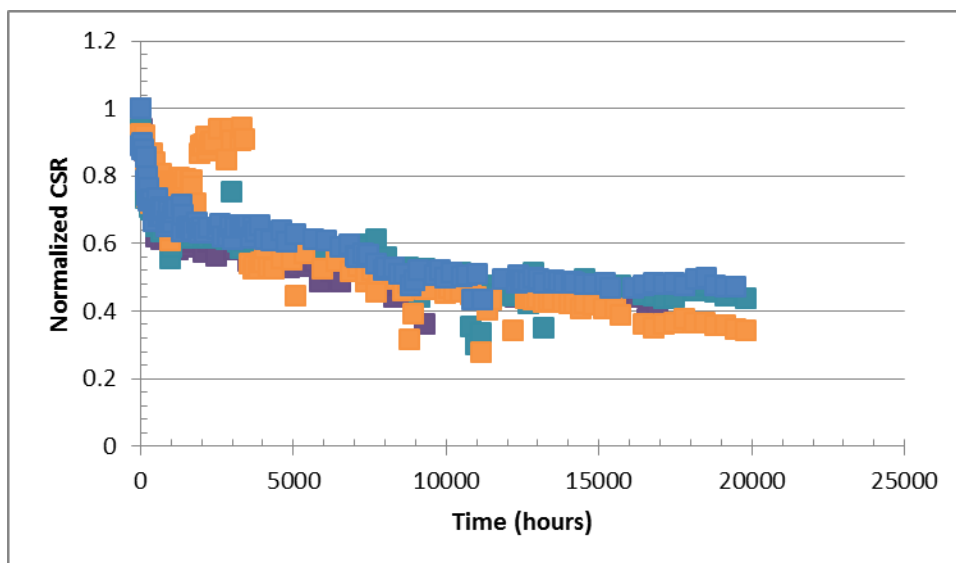


Figure A3: CSR values for GLT O-rings aged at 250 °F. These samples were removed from test after demonstrating no significant variation from the different pre-treatments.

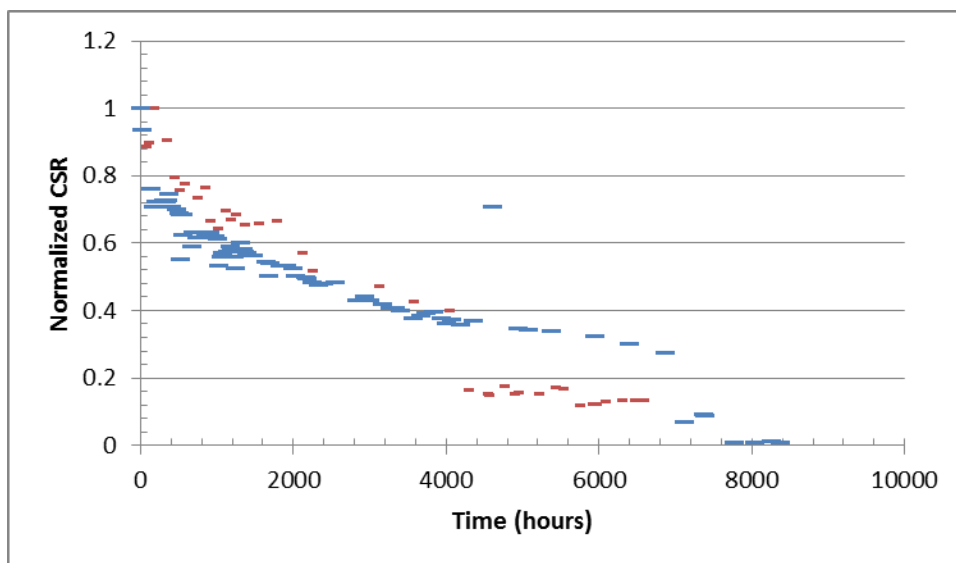


Figure A4: CSR values for GLT O-rings aged at 300 °F.

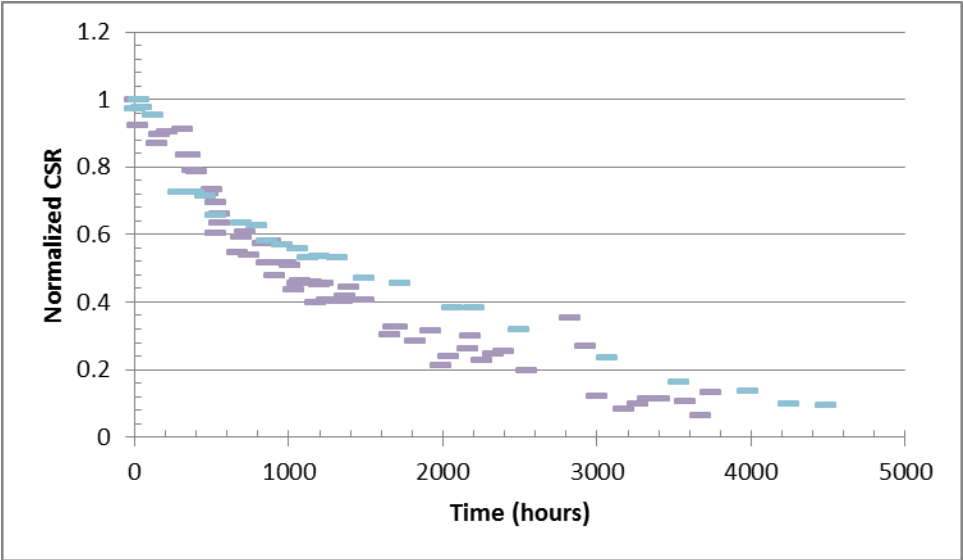


Figure A5: CSR values for GLT O-rings aged at 350 °F.

CSR Results for GLT-S Samples:

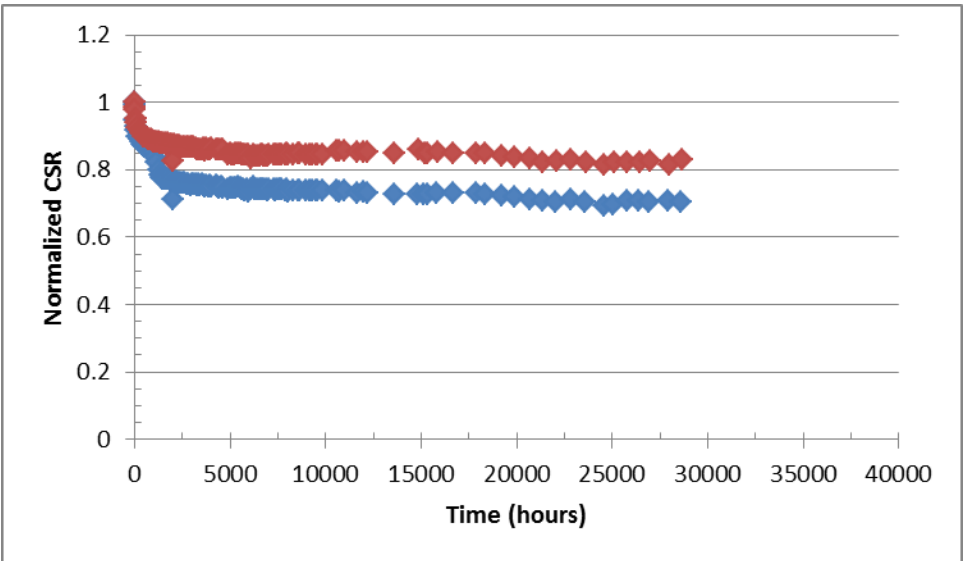


Figure A6: CSR values for GLT-S O-rings aged at 175 °F. These samples remain in test.

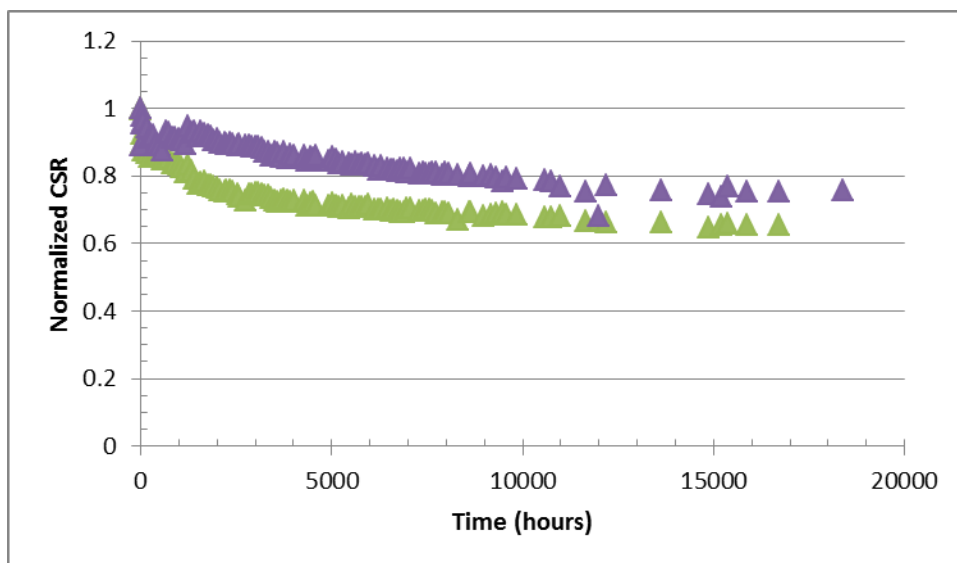


Figure A7: CSR values for GLT-S O-rings aged at 235 °F. Samples were terminated following a temperature excursion in April 2013.

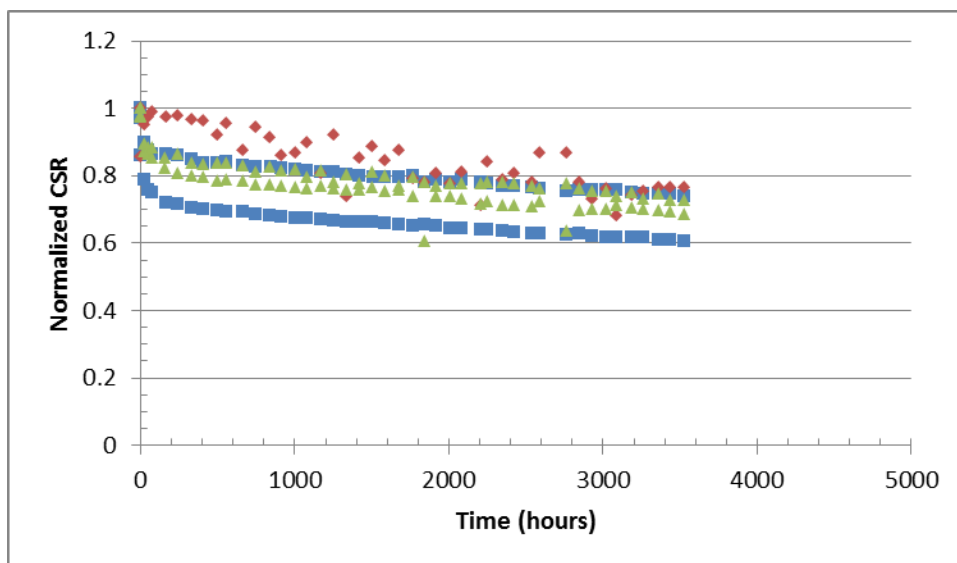


Figure A8: CSR values for GLT-S O-rings aged at 250 °F with no pretreatment, ■, stretched, ◆, and irradiated, ▲, showing similar CSR values between the three pretreatments. These samples were removed from test after demonstrating no significant variation from the different pretreatments.

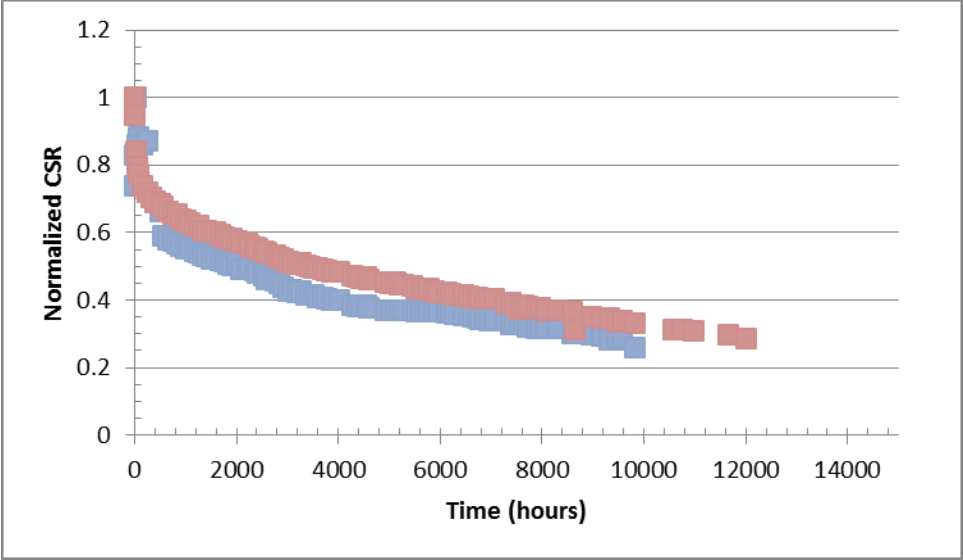


Figure A9: CSR values for GLT-S O-rings aged at 300 °F.

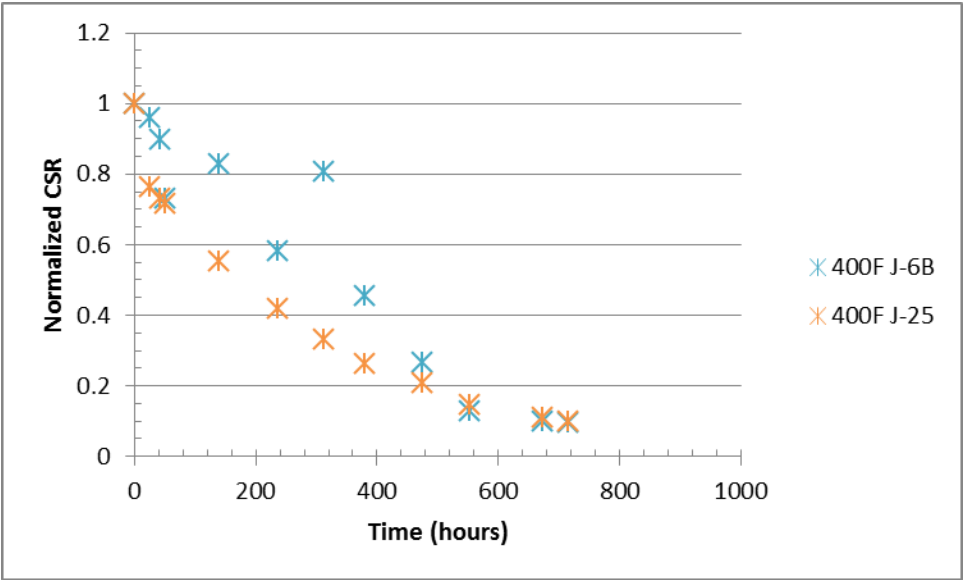


Figure A10: CSR values for GLT-S O-rings aged at 400 °F.

Distribution:

G.A. Abramczyk, 730-A
J.S. Bellamy, 730-A
G.T. Chandler, 773-A
W.L. Daugherty, 773-A
K.A. Dunn, 773-41A
B.A. Eberhard, 105-K
T.W. Griffin, 705-K
E.R. Hackney, 705-K
S.J. Hensel, 705-K
E. V. Henderson, 705-K
J. M. Jordan, 705-K
B. B. Kiflu, 705-K
M. D. Kranjc, 730-A
D.R. Leduc, 730-A
J.W. McEvoy, 707-C
A.J. McWilliams, 999-2W
T.E. Skidmore, 730-A
D.E. Welliver, 705-K
K.E. Zeigler, 773-41A
Records Administration (EDWS)