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STATUS REPORT FOR AGING STUDIES OF EPDM O-RING MATERIAL FOR THE H1616 SHIPPING PACKAGE

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SUMMARY

This is an interim status report for tasks carried out per Task Technical Plan SRNL-STI-2011-00506 [1]. A series of tasks/experiments are being performed at the Savannah River National Laboratory to monitor the aging performance of ethylene propylene diene monomer (EPDM) O-rings used in the H1616 shipping package. The data will support the technical basis to extend the annual maintenance of the EPDM O-rings in the H1616 shipping package and to predict the life of the seals at bounding service conditions.

Current expectations are that the O-rings will maintain a seal at bounding normal temperatures in service (152 °F) for at least 12 months. The baseline aging data review suggests that the EPDM O-rings are likely to retain significant mechanical properties and sealing force at bounding service temperatures to provide a service life of at least 2 years [2]. At lower, more realistic temperatures, longer service life is likely. Parallel compression stress relaxation and vessel leak test efforts are in progress to further validate this assessment and quantify a more realistic service life prediction.

The H1616 shipping package O-rings were evaluated for baseline property data as part of this test program. This was done to provide a basis for comparison of changes in material properties and performance parameters as a function of aging. This initial characterization was limited to physical and mechanical properties, namely hardness, thickness and tensile strength. These properties appear to be consistent with O-ring specifications.

Three H1616-1 Containment Vessels were placed in test conditions and are aging at temperatures ranging from 160 to 300 ºF. The vessels were Helium leak-tested initially and have been tested at periodic intervals after cooling to room temperature to determine if they meet the criterion of leaktightness defined in ANSI standard N14.5-97 (< 1E-07 std cc air/sec at room temperature). To date, no leak test failures have occurred. The cumulative time at temperature ranges from 174 days for the 300 ºF vessel to 189 days for the 160 ºF vessel as of 8/1/2012.

The compression stress-relaxation (CSR) behavior of H1616 shipping package O-rings is being evaluated to develop an aging model based on material properties. O-ring segments were initially aged at four temperatures (175 °F, 235 °F, 300 °F and 350 °F). These temperatures were selected to bound normal service temperatures and to challenge the seals within a reasonable aging period. Currently, samples aging at 300 ^{o}F and 350 ^{o}F have reached the mechanical failure point (end of life) which is defined in this study as 90% loss of initial sealing force. As a result, additional samples more recently began aging at ~270 ^{o}F to provide additional data for the aging model.

Aging and periodic leak testing of the full containment vessels, as well as CSR testing of O-ring segments is ongoing. Continued testing per the Task Technical Plan is recommended in order to validate the assumptions outlined in this status report and to quantify and validate the long-term performance of O-ring seals under actual service conditions.

BACKGROUND

The H1616 is a certified "Type B" package for the transport of radioactive tritium reservoirs by the Department of Defense (DoD), United Kingdom (UK) Atomic Weapons Establishment (AWE), and Authorized Users (Savannah River Site (SRS), Pantex), Figure 1 [3]. The H1616 packages were initially designed and produced by Sandia National Laboratory and were first certified in 1992. The packages are re-certified every 5 years by DOE/NNSA/Packaging Certification Division. There are two variations of the package, designated H1616-1 and H1616-2, which are of essentially the same design, except the H1616-2 has a slightly thicker containment vessel wall and has an option feature to include a getter between the two EPDM O-rings. Both H1616-1 and H1616-2 packages utilize the same O-ring seal design.

Aluminum packing material used to position the tritium reservoir is added to the containment vessel (CV) which is constructed of Type 304 stainless steel. The CV is placed inside a 16 gallon stainless steel drum (Figure 1). Two EPDM O-rings are housed between the CV lid and body and are used to seal each containment vessel. The seals are compressed nominally 25% (+/-3%) in concentric face seal grooves with a surface finish of 32 µinch or 32 RMS. No significant ID stretch is imposed, and no seal lubricant is used on the O-rings. All containers undergo annual re-verification testing, including containment vessel leak testing to verify leak-tightness to <1E-07 ref cc/sec air as per ANSI N14.5 [4].

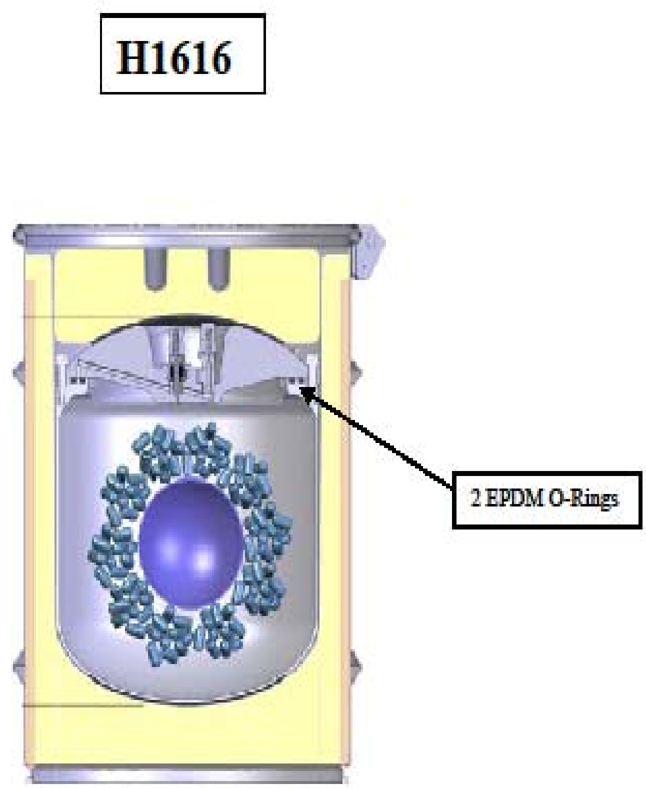

Figure 1. H1616 Container showing O-ring location

The O-rings used in the H1616 shipping package are fabricated in accordance with Sandia specification SS395668. This specification dictates material composition and curing conditions that a compounder/supplier must meet. The O-rings in the H1616-1 and the H1616-2 vessels are of slightly different major diameter. However, they have the same thickness, experience no stretch, and are compressed the same amount in service. They are made of the same composition, and see essentially the same environmental conditions. Therefore, only the H1616-1 vessel is being tested and the results of these tests can be considered equivalent for H1616-2 vessels.

The service temperature range for the H1616 vessel is -40 °F to 169 °F. The normal conditions of transport (NCT) temperature 169 °F is based on an ambient temperature of 100 °F with solar heating [3]. The maximum temperature at the flange closest to the O-rings is 152 °F with solar heating and 116 °F in the shade. The maximum temperature judged to be most applicable to the O-rings is 152 °F. Typical seal temperatures are expected to be lower.

Bounding radiation dose rates for the H1616 O-rings are not significant relative to seal performance [3].

The inner O-ring of the H1616-1 and H1616-2 without getter is credited as a containment boundary for transport purposes. For the H1616-2 with getter, the outer O-ring is credited as a containment boundary for transport purposes. The leak rate criterion for the EPDM seals is leaktight per ANSI N14.5, or < 1E-07 ref cc/sec air, which is performed at ambient temperature. A nitrogen or argon backfill gas is specified for the H1616 containment vessels. No lubricant is used for the O-rings.

SUMMARY OF BASELINE AGING DATA REVIEW

A literature/data review was performed on EPDM elastomer seals as part of this task. Focus was given to literature data believed to be the most relevant to sealing applications similar to the H1616 application. An abundance of aging data exists for EPDM elastomer in other applications, including outdoor weathering and seals in fluid service, but these were not considered appropriate for comparison. A summary of the literature/data review performed on EPDM O-rings is provided. Additional details are given in Reference 2.

Comparison of aging data for different EPDM compounds from multiple sources using varying approaches and parameters to define failure is complex. However, the collective data suggest that the H1616 EPDM O-rings can likely retain sufficient properties to remain leaktight for at least two years at or below 169 °F, the bounding NCT vessel temperature. Conservative interpretation of accelerated-aging data from examined references suggests possible lifetimes of at least 4 years at 76 °C (169 °F) and at least 6 years at 67 °C (152 °F), which is the peak O-ring temperature with solar heating. Even longer lifetimes should be possible at lower, more realistic service temperatures, as the NCT temperature assumes 100 °F ambient temperature with solar heating, neither of which are chronic conditions. Being a hydrocarbon-based elastomer, EPDM is known to be sensitive to thermally induced oxidation and therefore may exhibit a protective or inhibition period due to antioxidant protection. However, once the antioxidant is depleted or consumed, the polymer oxidation rate will likely increase, possibly resulting in an accelerated loss of mechanical properties and sealing force.

The antioxidant (Flectol® H) used in the H1616 O-ring compound is the same as used in some of the compounds evaluated in the data review. In at least one case (Reference 22 cited in the literature/data review), the composition of the H1616 O-ring compound is very similar except for the base polymer. In the H1616 compound, the base polymer is Nordel® 1470 whereas the base polymer in the reference compound (SR793B-80) in Reference 22 cited in the literature/data review was based on Nordel® 1440. The primary differences between these grades are that the 1440 grade has a Mooney viscosity of ~39 and a molecular weight of ~210,000, compared to having a Mooney viscosity of ~69 and a molecular weight of ~290,000 for the 1470 grade. The variation in long-term performance of compounds based on these polymers is difficult to predict based on available data.

The purpose of the work cited above (SRB793B-80 compound, Flectol® H antioxidant) was to predict weapon component seal performance at room temperature. In that case, the seals were predicted to have a mechanical lifetime of at least 150 years and possibly up to 2000 years at 25 °C (77 °F), even accounting for antioxidant protection and oxygen consumption rates (Figures 2 and 3). This prediction is based on Time-Temperature Superposition (TTS) analysis of CSR data, which is one of the test approaches being used with the H1616 O-rings. The advantage of TTS is that the data can be translated to any temperature of interest, assuming a constant activation energy and degradation mechanism. The CSR data showed that a ~90% loss in sealing force (F/Fo<0.1) was observed after ~580 days (1.6 years) at 111 °C (232 °F), which is notably very close to the 235 °F aging temperature currently being used in the SRNL study on the H1616 O-rings.

From the same data, a shift factor of ~0.035 is estimated for the bounding H1616 O-ring temperature of 152 °F (340K, 1/T = 0.00294) from Figure 2, increasing the estimated service life to 580/0.035 days or ~ 45 years. Similarly, a shift factor of ~0.08 at the NCT temperature of 169 °F (349K, 1/T=0.00287) provides a service life estimate of 580/0.08 days or approximately 19 years, assuming a constant activation energy of 82 kJ/mol. While these data certainly indicate that the H1616 seals should be able to maintain similar functionality for at least 2 years or more, there are a couple of primary limitations of these data to consider. Notably, it is unknown whether the variation in the base polymer (Nordel 1470 in the H1616 compound and Nordel® 1440 in the SR793B-80 compound) impacts the aging behavior. In addition, the reference CSR data (Figure 3) are limited to only two temperatures (111 °C, 125 °C). The use of only two temperatures is insufficient to verify the assumption that the activation energy for CSR behavior remains constant, limiting the current usefulness of these data.

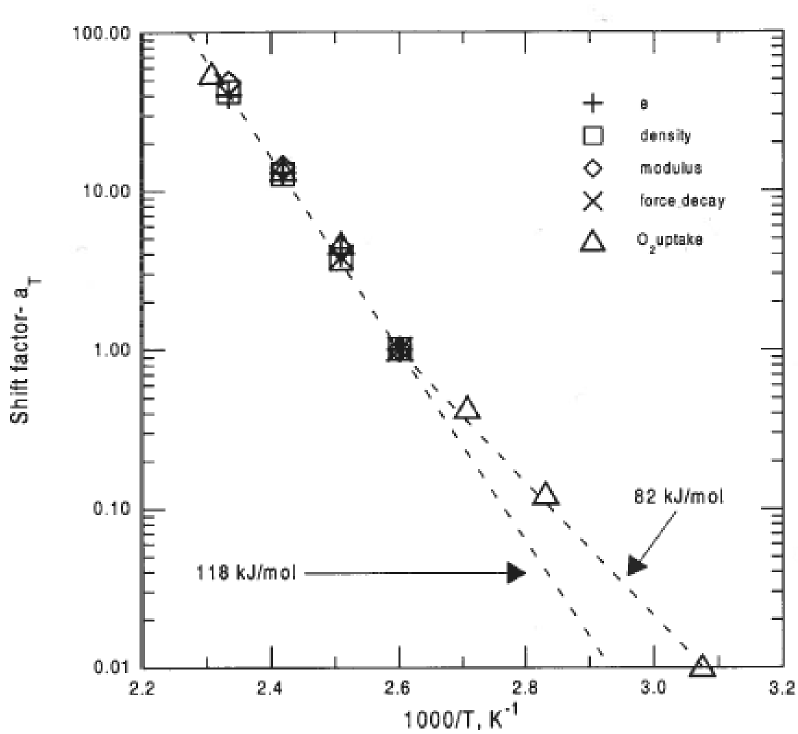

Figure 2. Arrhenius plot of shift factors for EPDM vs. inverse absolute temperature [Reference 22 cited in the literature/data review]

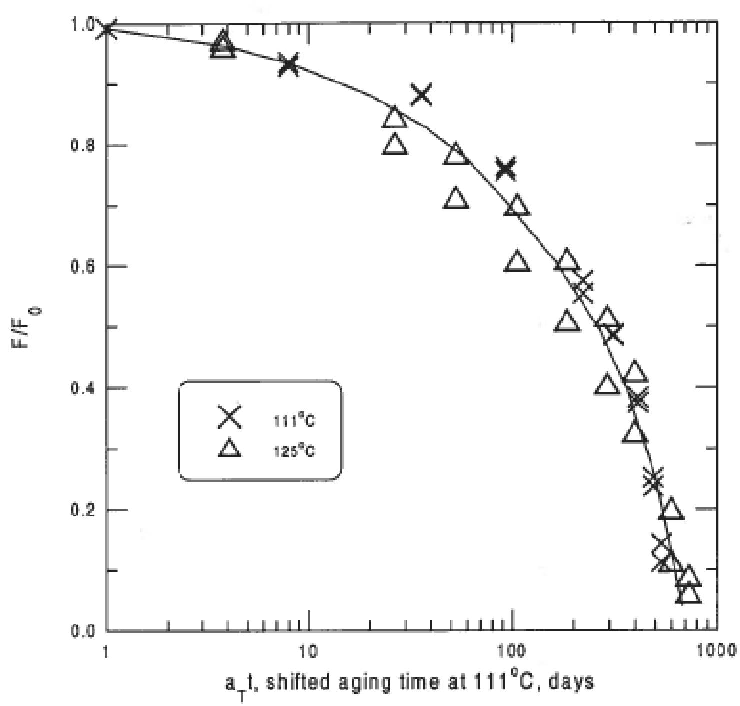

Figure 3. Time-temperature superposition of CSR data for EPDM (SR793B-80) at a reference temperature of 111 °C (232 °F) [Reference 22 cited in the literature/data review]

Another reference (Reference 21 cited in the literature/data review) strongly suggests that mechanical properties and sealing performance will be highly retained at the bounding temperatures, 169 °F (76 °C) in the H1616 shipping package for at least 2 years. A retained sealing force value of ~60-65% is estimated at this temperature for a 2 year period, based on data for a slightly different compound at aging temperatures of 60, 70 and 80 °C. These values fall within the range of acceptable behavior for standard seal designs.

A summary of the EPDM seal lifetimes observed or predicted at various temperatures from different references is given in Figure 4. Direct comparison is difficult as the basis for performance or failure in each reference varies. Data points represented by solid symbols (References 13, 15 and 21 cited in the literature/data review) are not actual failures as defined by CSR or leakage, but represent samples removed from test prior to failure.

The trend line shown in Figure 4 (which is a lower bound to all failure data) is intended to show reasonable and conservative lifetimes that might be predicted from the available data. Actual lifetimes could vary. At 76 °C (169 °F), the trend line suggests a lifetime of at least 4.2 years. At 67 °C (152 °F) the trend line suggests a lifetime of ~ 5.8 years. At 47 °C (116 °F) (max O-ring temperature in shade), the estimated lifetime is increased to about 19 years. In absence of solar heating and ambient temperatures greater than 100 °F, the 116 °F O-ring temperature is likely more realistic (but still bounding) for typical exposure.

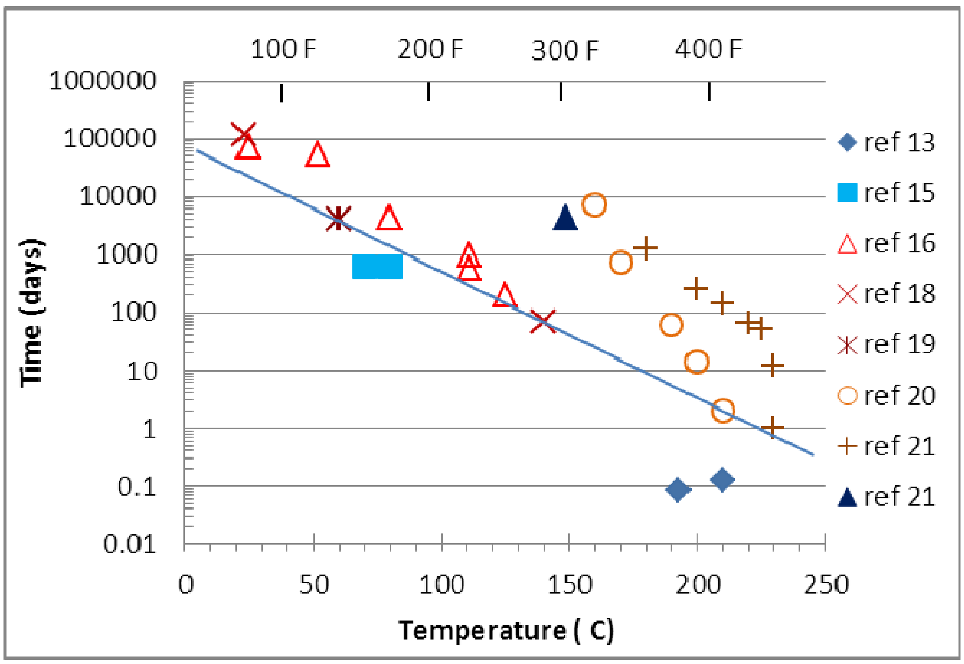

Figure 4. EPDM O-ring lifetime data from literature references

The data review was primarily focused on long-term performance of EPDM seals at elevated temperatures consistent with NCT. The performance of aged seals at Hypothetical Accident Conditions (HAC) (post-fire or low temperature) or other extreme conditions is not part of the current SRNL study. With a low glass transition temperature (-58 °F or lower), EPDM seals have significant margin at -40 °F such that performance even after significant aging is likely acceptable. However, the ability of aged seals (of any elastomer) to maintain a leaktight seal at -

40 °F has not been widely studied. This is not considered a major concern for the H1616 package since low temperature conditions are not anticipated in service and would not be expected to be a chronic condition. However, testing of seals at extreme conditions after aging may be necessary to establish acceptable limits.

Despite the favorable O-ring performance identified in the literature review, there is no substitute for real-time aging at realistic or bounding conditions. Therefore, the periodic examination and testing of actual seals from a select group of H1616 shipping packages aged at bounding conditions are being used to validate assumptions and make predictions based on accelerated-aging methodology. Such examinations cannot predict time to failure until failure is actually observed, but advance notice of degradation could be provided. A description of these tests is included in this report.

EXPERIMENTAL APPROACH

Baseline O-Ring Characterization

The H1616 O-ring compound formulation is given in Table 1. Conformance of the H1616 compound to any particular ASTM or SAE/AMS standard is unknown.

Table 1. H1616 EPDM seal compound SS#395668

Formulation, phr*		
	Nordel 1470	100
	Zic Stik '85'	5
	N-990 carbon black	40
	N-539 carbon black	25
	DiCup 40C	12
	Flectol H antioxidant	2
	SR-350 Sartomer	10
Physical Properties		
	Thickness, inches	0.21 +/- 0.005
Mechanical Properties		
	Hardness, Shore A	78 +/- 5
	Tensile Strength, psi	1200 minimum
	Elongation, percent	100 minimum

* phr – parts per hundred rubber (by weight)

Confirmatory measurements were performed on a limited number of O-ring samples to verify the O-ring physical and mechanical properties are consistent with existing published vendor data. Thickness measurements were verified with a calibrated Snap Gage taken at four equally-spaced points along the circumference in both the top-to-bottom orientation and the side-to-side orientation. Hardness measurements were verified with a calibrated Durometer Shore Hardness tester measured on the top of the O-ring at 5 points along the circumference using two scales, Durometer Shore A (per Specification) and Durometer Shore M (for comparison). Durometer Shore M scale is the industry standard and ASTM accepted scale for a round O-ring sample less than 0.25 inch thick.

H1616 Vessel Aging and Leak Testing

Vessel aging and leak tests are being performed using three full-size containment vessels. Since aging at room temperature is not expected to produce leak failure in the time allotted for testing (two years), these test CVs are being kept at conditions that bound operating temperatures with the intent to drive the O-rings to failure within reasonable test periods. This approach uses actual vessels and the acceptance criterion (leaktightness) which is the most relevant for performance. Failure of the CV seal will be a leak rate greater than the leaktight criterion of 1E-07 cc/sec dry air at 25 ºC and 1 atm pressure differential. The vessels are being heated externally such that O-ring temperatures are maintained at either 160 ^{o}F, 235 ^{o}F, or 300 ^{o}F. The use of three temperatures allows extrapolation of results to support life prediction model development. These temperatures are based on bounding service temperatures and bounding "continuous" temperature ratings as indicated from typical EPDM compounds. Again, the 160 ^{o}F temperature is bounding for normal service as it accounts for solar heating superimposed on a 100 ^{o}F ambient temperature, neither of which are chronic conditions.

Each CV sits in a round metal pan within a test stand (Figure 5). Two 6 inch x 20 inch Watlow® heaters, wired in series, are wrapped around the upper half of the vessel. These heaters provide 300 watts (2.5 amps) of heating to the vessel. 24 layers of aluminum foil are placed between the heater and the vessel OD to accommodate the 40" heater length without overlap. The foil, heaters and 1 layer of silica insulation are secured to the vessel with band clamps. Additional silica insulation is added to the side, top and bottom of the vessel to help regulate vessel temperature. Additional band clamps hold the side insulation in place (Figure 6). Ring supports, bolts, and braces were fabricated to hold the vessel secure in the stand.

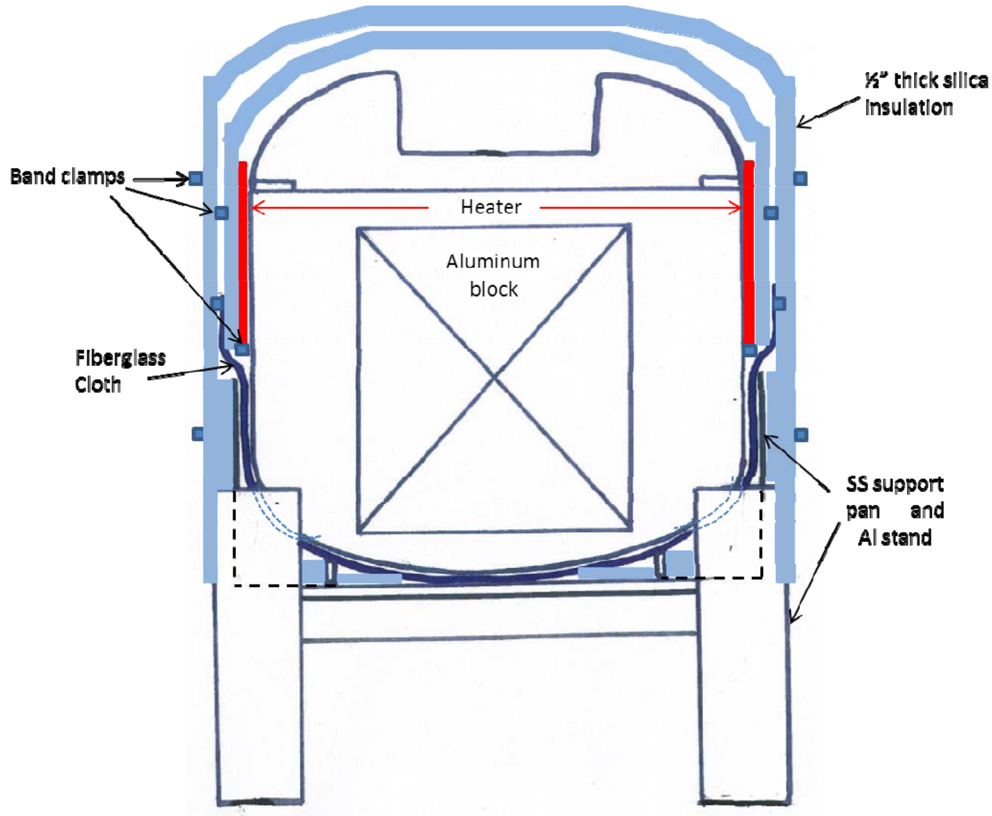

Figure 5. Cross section sketch of vessel and insulation arrangement.

Figure 6. Containment vessel test setup

Four thermocouples are placed at 90 degree intervals between the aluminum foil and the outside of vessel, approximately 1.5 inches from the top surface of the CV lid. Thermocouples were verified with a calibrated Dry Well Calibrator and thermocouple reader. The heaters are controlled by a LabView® software program. One thermocouple from each vessel is used to control the heater, while an additional two provide additional assurance that the vessel is heating uniformly. The fourth thermocouple provides a signal to an over-temperature controller which prevents an unplanned thermal excursion.

Tritium Packaging Operations replaced the sample valve O-ring, the sample fitting O-ring, and the hex head plug O-ring on all three vessels after the trial runs and before new CV O-rings were installed to begin testing. The new CV inner and outer O-rings were installed by SRNL.

A thin strip of 0.005 inch thick stainless steel shim is placed on the vessel flange between the O-rings to prevent a possible metal-to-metal seal from masking the leak behavior of the O-rings. Initial tests were performed to show permeation (gradual increase in detector response over time) to prove helium access to/through the O-rings. In addition, vessel O-ring temperature / vessel gradient heat up trials were performed to determine the correct temperature control settings, as shown in Table 2.

Table 2. Vessel O-ring/control Temperature Comparison

Vessel Lid ID	**Desired O-ring Aging Temperature (^{o}F)**	**Vessel Exterior Control Temperature (^{o}F)**
TDA-13774-G92	160	165
TDA-11296-D92	235	241
TDA-13896-G92	300	313

An aluminum block (8 inches diameter x 10 inches long) was placed inside each vessel to reduce the amount of gas space and facilitate leak testing.

Argon (at least 99.99%) is used as a backfill gas. Leak tests are performed periodically, after the vessel has cooled down to room temperature. Leaktightness is verified per ANSI N14.7 with a hood test performed by a certified Level II Leak Test Specialist. The vessel is bagged, and the vessel interior and exterior are evacuated and backfilled with helium (Figure 7). A helium mass spectrometer leak detector is connected to the test volume between the two O-rings, and will detect leakage from either O-ring. In the event of detected leakage, the vessel connections can be re-configured to test each O-ring individually.

Figure 7. Hood test on containment vessel

The test CV's are opened periodically in conjunction with leak testing keeping the same O-rings in place. This is not expected to adversely impact the results of these tests, and will actually prevent artificial "sticking" of the O-rings, which could provide a false positive result. Additionally, it provides a more realistic representation of actual in-service practice.

Compression Stress Relaxation Testing

Compression stress relaxation (CSR) tests are being performed on the H1616 O-rings at several aging temperatures to develop an aging model based on sealing force decay. In the absence of fluid or significant radiation exposure, thermal aging and sealing force decay is expected to be the most relevant degradation mechanism for the H1616 O-rings. CSR tests are a common industry method for evaluating the mechanical performance of seals over time at various aging temperatures. CSR tests are being performed per ASTM D6147, using the periodic measurement approach.

A single H1616 O-ring was carefully cut into several segments ~0.75" long, with three segments placed into each CSR jig (Shawbury-Wallace type). The CSR jigs were initially developed to compress large disc samples, but several studies have shown that diffusion-limited oxidation effects are possible with larger samples, and disc samples do not ideally represent actual O-ring behavior, even if made of the same elastomer compound [5]. Three segments were selected to balance the contact area of the compression platens and the surface area of the exposed material. The segments were then compressed approximately 25% from 0.21” cross-section thickness to ~ 0.1575” as in the H1616 design. A fabricated shim was used to verify compression.

The jigs with compressed samples were placed into aging ovens, with three jigs per temperature for repeatability. Aging temperatures were initially selected as 175 °F, 235 °F, 300 °F and 350 °F. These aging temperatures were selected to bound expected normal service temperatures and to challenge the seals within a reasonable aging period. Prior to compressing samples in the jigs, the break force for each jig is determined using the relaxometer. Each jig has its own break force, which is typically in the range of 4-10N. The break force is subtracted from later total force measurements to give sealing force measurements only. Upon initial compression but prior to aging, the compressed jigs are placed into the relaxometer to obtain a baseline sealing force measurement at room temperature. During aging, the CSR jigs are periodically removed from the aging ovens and placed in the relaxometer (Figure 8) for measurement of sealing force, which is the force required to deflect the compressed seals approximately 0.0001-0.0002”. As the elastomer relaxes and ages over time, this counterforce value is reduced. During periodic sealing force measurements, five sequential readings or runs are taken, with time and temperature of the jig monitored. Measurements are taken as quickly as possible to capture the sealing force at the aging temperature, rather than waiting for the fixture to cool to room temperature. CSR values can be taken at any temperature, but should be taken as close to the aging temperature as possible for direct comparisons and interpretation of sealing behavior at the actual temperature of interest.

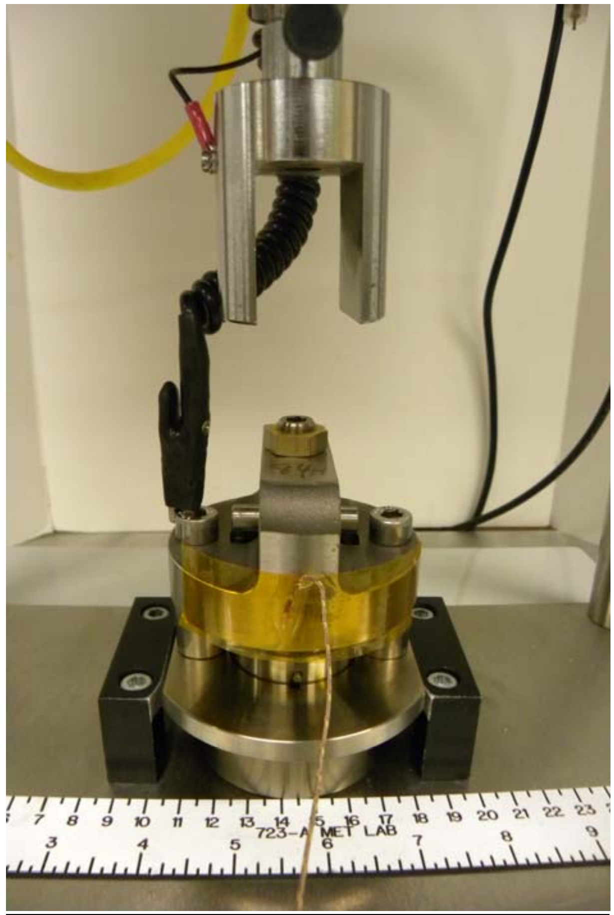

Figure 8. CSR jig in relaxometer for sealing force measurement (thermocouple wire is attached to the jig during aging and CSR measurements)

The main goal of the CSR tests is to age the seals to the point of mechanical failure at multiple temperatures, which allows development of an aging model based on Arrhenius theory and TTS of the collective data. Ideally, failure will be reached at all aging temperatures within the planned aging period (2 years) for model development. The true time to failure for elastomeric seals can vary with the design, function of the seal as well as the parameter being monitored. For CSR testing, a 90% loss in sealing force (F/F_o=0.10) is often used as the mechanical failure point for elastomeric seals in typical designs, and this parameter has been used in critical seal applications such as nuclear weapon components [6]. It is important to note that CSR failure does not inherently mean that the seal is no longer leaktight. The use of 90% sealing force loss leaves 10% of initial sealing force remaining for margin, which is expected to be sufficient for most static seal designs. Seals have been known to remain leaktight well beyond the 90% loss threshold, with some seals being able to remain leaktight even at 100% loss in sealing force. The relationship between CSR and leak rate has not be established for this design and was not part of the scope for this task.

The advantage of the TTS principle is that all of the data can be used in the aging model, rather than using only a few time to failure data points. Another advantage of the TTS principle is that if the same degradation mechanism exists over the full aging temperature range and at the expected service temperatures, the aging model can be translated to any temperature of interest.

RESULTS / DISCUSSION

Baseline O-Ring Characterization

Confirmatory measurements on twelve new O-rings were performed to verify the material properties are consistent with existing published vendor data (Table 3). All O-ring thickness measurements were within the specified range of 0.21 +/- 0.005 inches. All O-ring hardness measurements were within the specified range of 78 +/- 5 A scale. Both the thickness and hardness measurements are non-destructive.

Since the tensile strength and elongation tests are destructive, a limited number of O-rings were tested using the Instron Tensile Tester. The O-rings tested were within the specified requirements of greater than 1200 psi Tensile Strength, and greater than 100 percent elongation.

Table 3. Baseline confirmatory measurements of O-rings

Sample ID:	Average Thickness (inches) (top – bottom)	Average Thickness (inches) (side – side)	Average Hardness (A)	Average Hardness (M)	Tensile Strength (psi)	Elongation (%)
1 -1 Outer	0.2090	0.2120	80	80	2267	335
1 -1 Inner	0.2076	0.2120	80	80	2017	294
2 -1 Outer	0.2090	0.2123	80	80		
2 -1 Inner	0.2085	0.2113	80	79		
3 -1 Outer	0.2096	0.2123	80	80		
3 -1 Inner	0.2088	0.2121	79	79		
4 -1 Outer	0.2091	0.2118	80	77		
4 -1 Inner	0.2089	0.2120	80	81		
5 -1 Outer	0.2093	0.2113	80	78	2116	392
5 -1 Inner	0.2086	0.2126	80	79	2137	340
6 -1 Outer	0.2099	0.2123	80	79		
6 -1 Inner	0.2089	0.2130	80	79		

H1616 Vessel Aging and Leak Testing

Three containment vessels which have seen actual service were transferred to SRNL.

Container #	Container Body #	Target Aging Temp ^{o}F
13896	13772	300
11296	11274	235
13774	13784	160

Initial baseline leak tests were performed on each vessel by a certified Level II Leak Test Specialist on 1/3/12. Heating of containment vessels began at 0830 on 1/4/12 and all three reached target temperature the same day. Leak tests were performed on each vessel after one month and at varying intervals thereafter. All three vessels have passed each leak test performed to date, after which they were backfilled with Argon (99.999%) and returned to test temperature. (Table 4)

Table 4. Containment vessel leak tests

Temp (F)	**Days at temp Pass/Fail**									
300	0 – pass	25- pass	47 – pass	64 – pass	81 – pass	99 – pass	127 – pass	145 – pass	161 – pass	174 – pass
235	0 – pass	25- pass				108 – pass				189 – pass
160	0 – pass	25- pass				108 – pass				

On 2/29/12 (after 47 days at temperature) the CV lid for the vessel at 300 ^{o}F was removed, and a visual inspection was performed on the O-rings. A small amount of sticking was observed and an oily residue was observed on the O-rings and in the O-ring grooves. The O-rings and the O-ring grooves were cleaned using isopropyl alcohol to remove the residue, and replaced in the vessel. Another leak test was performed to verify that leaktightness was not compromised by the inspection. During each subsequent leak test cycle, the lid to the 300 ^{o}F vessel was removed, a visual examination of the O-rings was performed, and O-ring hardness was measured (Table 5). During the April 11, 2012 visual inspection (after 81 days at temperature), it was noted that the inner and outer O-rings were beginning to “flatten” and lose their round shape. Visual and hardness tests were performed on the O-rings in the 235 ^{o}F vessel on 5/2/12 (after 108 days at temperature) and on 8/1/12 (after 189 days at temperature), and no sticking was observed. Based on these observations, the 160 ^{o}F vessel has not yet been opened for visual inspection of the O-rings.

To date, all leak tests performed have been acceptable as defined by ANSI N14.5 [4] (< 1E-07 ref cc/sec air).

Table 5. O-rings in vessels (aging in Argon) – hardness

300 ^{o}F

Outer		Inner	
Time @ temp	Hardness (Durometer M)	Time @ temp	Hardness (Durometer M)
0 days	80.0	0 days	78.9
64 days	78.3	64 days	76.9
81 days	76.8	81 days	74
99 days	78.7	99 days	76.5
127 days	78.8	127 days	79.3
145 days	78.8	145 days	78.3
161 days	77.5	161 days	78.7
174 days	79.7	174 days	79.8

235 ^{o}F

Outer		Inner	
Time @ temp	Hardness (Durometer M)	Time @ temp	Hardness (Durometer M)
0 days	80.0	0 days	78.6
108 days	79.2	108 days	78.6
189 days	80.8	189 days	78.2

160 ^{o}F

Outer		Inner	
Time @ temp	Hardness (Durometer M)	Time @ temp	Hardness (Durometer M)
0 days	77.2	0 days	81.4
~ 300 days	due 11/2012	~ 300 days	due 11/2012

Compression Stress Relaxation Testing

CSR jigs with compressed H1616 O-ring segments were placed in aging ovens at the following temperatures in November 2011: 175 °F, 235 °F, 300 °F and 350 °F. CSR measurements were recorded approximately every 30 days. Initial sealing force values range from 512 to 644N for all CSR jigs. Variation in the initial sealing force is attributed to jig variability and to the exact degree of compression achieved on each segment or set of segments in each jig. A shim fabricated to 25% of nominal thickness (0.1575") was used to check the degree of compression in each jig. All O-ring segments came from a single O-ring so thickness for each segment was assumed to be constant, but this was not verified.

In late January 2012, some O-ring segments in at least one jig aging at 350 ^{o}F were observed to be split vertically across the compressed cross-section. Sealing force values were obtained for other jigs at 350 ^{o}F so additional aging was allowed to continue. The 350 °F CSR O-ring segments reached an end-of-life condition (90% loss in initial sealing force) in February 2012, and O-ring segments that were aging at 300 ^{o}F reached an end-of-life condition in June 2012. Cracked EPDM samples after aging at 350 °F for approximately 3 months are shown in Figure 9. The cracks were observed to run vertically relative to the installed or compressed orientation in the CSR jigs, consistent with internal stress. Residue on CSR jig platens after aging at 350 °F is shown in Figure 10.

Figure 9. Cracked O-ring samples aged at 350 °F for approximately 3 months

Figure 10. Residue on compression platens from samples aged at 350 °F.

At the end of life, samples aging at 300 °F did not exhibit signs of cracking as observed at 350 °F. The higher 350 °F aging temperature is quite high for EPDM elastomer, which is normally rated for service temperatures of 300 °F or less, with long-term or continuous service temperature limits of 250 °F being more typical.

As a result of early failures at 350 °F, additional O-ring segments from the same initial O-ring were cut and placed into the same CSR jigs from the 350 °F tests and aged at 270 °F to maximize Time Temperature Superposition data. These jigs began aging at 270 °F in April 2012. The 270 °F temperature was selected as an intermediate temperature between the 300 °F and 235 °F aging temperatures for convenience.

Sealing force decay curves (CSR curves) for the H1616 O-ring segments at aging temperatures are given in Figure 11. The data shown are for the average of three jigs at each temperature. Minor variation is observed with each jig at a given temperature but the overall behavior of each jig is relatively similar. Aging at the 270 °F temperature began more recently, so the aging time is lagging other temperatures. The loss of sealing force at the lower aging temperatures (175 °F and 235 °F) is less severe with end of life (90% loss or higher) observed much sooner at the higher aging temperatures (300 °F and 350 °F) as expected.

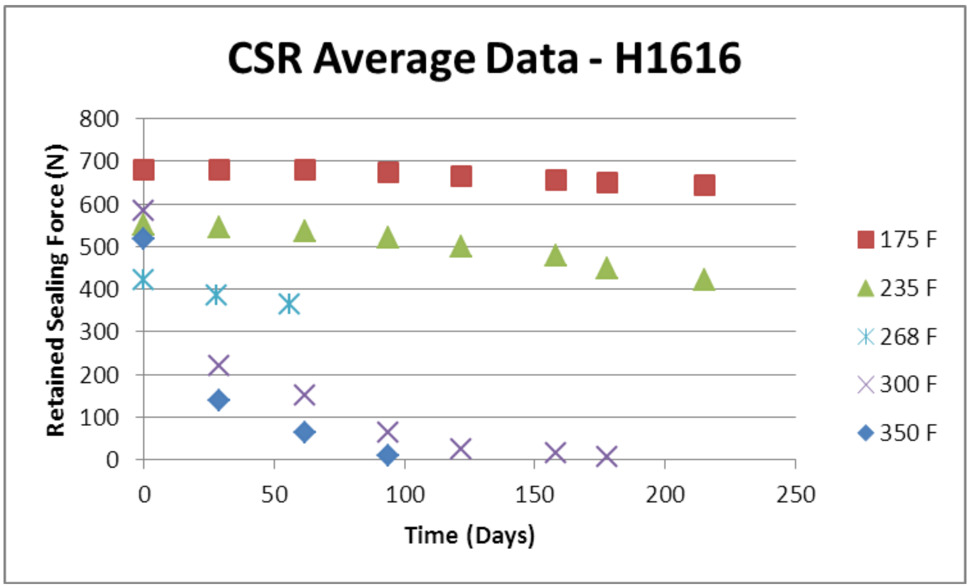

Figure 11. CSR average data for the H1616 O-ring segments at various aging temperatures

Using the TTS principle, the raw CSR data can be superposed into a "master" curve using shift factors (aT) empirically determined to achieve the best curve fit possible. However, at this time, the end of life condition has only been reached at two temperatures (300 and 350 °F), therefore aging model development is premature. In addition, the cracking behavior observed at 350 °F suggests a possible different degradation mechanism which may not be applicable to service temperatures. Inclusion of these data could give erroneous life prediction results. However, the CSR data to date suggests that the seals can likely tolerate at least 2 years at service temperatures of 169 °F (76 °C) and below unless a significant increase in relaxation occurs over the anticipated duration of the test program.

Additional O-ring aging mechanical tests

A significant difference in O-ring degradation and aging behavior was observed between the CSR samples being aged in air and the O-rings aging in the vessels with argon backfill. It was suspected that the argon backfill prevents oxidation of the O-rings, thereby significantly extending the useful service life. As a check on this effect, two sets of O-ring segments were placed in each of the CSR furnaces in order to track changes in the O-ring tensile properties and hardness (Table 6a and 6b). One set of these samples was exposed to air, while a second set was placed inside an enclosed container backfilled with argon. These containers consist of stainless steel tubing sealed with Swagelok connectors, to allow periodic opening for testing. This test approach provides common test parameters that can be verified following aging in each atmosphere (argon, air) for a direct identification of the impact of oxidation.

In addition to the two sets of samples, six baseline (unaged) O-ring samples were tested: tensile strength ranged from 1637 to ≥ 2267 psi, while elongation ranged from 246 to ≥413 percent. Additional values are presented in Table 6b. Aging continues on O-ring segments at 235 °F and at 175 °F.

Baseline tensile samples were initially tested with a grip arrangement in which the O-ring segment passed over a rounded mandrel to reduce the stress where the sample was gripped. It was observed that samples aged at the higher temperatures quickly became too stiff to wrap around the mandrel without breaking. Therefore, an alternate arrangement was developed and used for all aged samples. The ends of the sample were placed within multiple layers of thin flexible tube, and then placed between the faces of a flat grip. The tube layers provide isolation from the rough grip face, while generating enough friction against the sample to minimize slipping. This arrangement allowed testing of aged samples without forcing the sample to flex prior to loading. It was observed that the samples which had become stiff often broke as soon as the applied load caused any appreciable straightening of the sample. The sample also typically developed numerous circumferential cracks during the tensile test.

When a tensile sample broke in or adjacent to the grip, it was identified as potentially not achieving the true maximum load and elongation. If sufficient length remained, the sample was re-tested. In addition, a few samples slipped out of the grip during test – these samples were also re-tested. In either case, it is possible that the initial test produced cracks or other damage in the aged samples and created a bias in the subsequent results toward a lower strength / elongation. In such a case, the maximum strength and elongation values recorded for any trial on a given sample should be considered a lower bound for the actual properties of that sample and are the only values recorded in Table 6b for that sample.

Table 6a. O-ring segments in air vs. Argon – hardness properties

350 °F			
Air		Argon	
Time @ temp	Hardness (M)	Time @ temp	Hardness (M)
0 weeks	75.4	0 weeks	80.0
		1 week	79, 78.2
2 weeks	88.8, 88.8	2 weeks	78.2, 78.0
3 weeks	91.3	3 weeks	77.2
4 weeks	93.2, 94.8	4 weeks	78.2
5 weeks	95.6	5 weeks	78.0
6 weeks	96.0	6 weeks	78.3
7 weeks	96.8	7 weeks	78.0
8 weeks	96.7	8 weeks	

300 °F			
Air		Argon	
Time @ temp	Hardness (M)	Time @ temp	Hardness (M)
0 weeks	78.5	0 weeks	80
2 weeks		2 weeks	78.2, 79.5
3 weeks		3 weeks	78.3, 79.2
4 weeks	89.6, 89.6	4 weeks	79, 78.5
5 weeks		5 weeks	78.7
6 weeks		6 weeks	78.9
7 weeks		7 weeks	79.0
8 weeks	95.0	8 weeks	80.0

235 °F			
Air		Argon	
Time @ temp	Hardness (M)	Time @ temp	Hardness (M)
0 months	79.1	0 months	78.6
1 months		1 months	77.3, 76.8
2 months	80.0, 79.8	2 months	
3 months	81.3, 81.0	3 months	78.2, 77.2
4 months	81.6, 82.0	4 months	*
5 months	82.7, 82.6	5 months	*
6 months	*	6 months	*

175 °F			
Air		Argon	
Time @ temp	Hardness (M)	Time @ temp	Hardness (M)
0 months	78.3	0 months	78.6
1 months		1 months	78, 78.8
2 months	77.7, 79	2 months	77.7, 78.0
3 months	78.7, 78.7	3 months	78.8, 79.0
4 months	78.0, 78.3	4 months	*
5 months	79.3, 79.5	5 months	*
6 months	*	6 months	*
12 months	*	12 months	*

*Test in progress

Table 6b. O-ring segments in air vs Argon – tensile and elongation properties

350 °F					
Air			Argon		
Time @ temp	Tensile Strength (psi)	Elongation (%)	Time @ temp	Tensile Strength (psi)	Elongation (%)
0 weeks	1668	286	0 weeks	≥ 2267	≥ 335
2 weeks	≥ 301	≥ 45	2 weeks	2238	331
4 weeks	≥ 335	≥ 14	4 weeks	2118	297
0 weeks	≥ 2267	≥ 335			
2 weeks	≥ 292	≥ 21			
4 weeks	≥ 614	≥ 14			

300 °F					
Air			Argon		
Time @ temp	Tensile Strength (psi)	Elongation (%)	Time @ temp	Tensile Strength (psi)	Elongation (%)
0 weeks	≥ 1773	≥ 413	0 weeks	≥ 2267	≥ 335
4 weeks	315	20	4 weeks	2044	267
8 weeks	≥ 461	≥ 13	9 weeks	2018	235
235 °F					
Air			Argon		
Time @ temp	Tensile Strength (psi)	Elongation (%)	Time @ temp	Tensile Strength (psi)	Elongation (%)
0 months	1637	246	0 months	2137	340
3 months	1799	≥ 247	3 months	1999	282
6 months	*	*	6 months	*	*
175 °F					
Air			Argon		
Time @ temp	Tensile Strength (psi)	Elongation (%)	Time @ temp	Tensile Strength (psi)	Elongation (%)
0 months	≥ 1824	≥ 377	0 months	2137	340
6 months	*	*	6 months	*	*
12 months	*	*	12 months	*	*

*Test in progress

CONCLUSIONS

Review of relevant aging data for EPDM elastomers suggests that the H1616 O-rings should remain functional for at least 2 years at bounding conditions. Direct comparison of data from various references with different compounds, approaches and failure criteria is difficult. However, a conservative interpretation of the available data suggests that the H1616 O-rings can have lifetimes of nearly 6 years at 152 °F (maximum temperature at the flange closest to the O-rings with solar heating). This temperature is conservative relative to typical O-ring temperatures in service.

Baseline characterization was limited to physical and mechanical properties, namely hardness, thickness and tensile strength. These properties appear to be consistent with O-ring specifications.

O-rings installed in the H1616 vessels have been aged for 174 days at 300 °F and for 189 days at 235 °F and 160 °F as of August 1, 2012. Leak testing has demonstrated acceptable leak-tight performance at these durations for the 300 °F and 235 °F vessels. Leak tightness of the 160 °F vessel will be verified at a future date. This testing will continue, in order to demonstrate increasingly longer aging intervals.

CSR test samples aging at 300 and 350 ^{o}F in an air environment have reached a failure condition after 150 and 90 days, respectively. Early stress-relaxation data suggest that the O-rings will retain significant sealing force at temperatures below 174 ^{o}F (79 ^{o}C), which is bounding to the NCT temperature, unless significant relaxation occurs during the remaining test duration. Once samples aging at a third temperature reach a failure condition, a preliminary aging model can be constructed to predict the O-ring behavior at service conditions. Samples were more recently added at 270 ^{o}F for model development.

While the baseline literature review and data obtained to date provide a favorable assessment regarding a potential 2 year service life at bounding conditions, the ultimate demonstration of service life is derived from the demonstrated successful continued leaktightness of a vessel at bounding service conditions for the service period of interest. Accordingly, aging and periodic leak testing of the full containment vessels, as well as Compression Stress Relaxation (CSR) testing of O-ring segments are ongoing. Continued testing is recommended for H1616 containment vessels and CSR jig for at least 2 years at temperature (as described in the Task Technical Plan) in order to validate the assumptions outlined in this status report and to assess the long-term performance of O-ring seals under actual service conditions.

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