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REVIEW OF AGING DATA ON EPDM O-RINGS IN THE H1616 SHIPPING PACKAGE

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1.0 EXECUTIVE SUMMARY

Currently, all H1616 shipping package containers undergo annual re-verification testing, including containment vessel leak testing to verify leak-tightness ($<1 \times 10^{-7}$ ref cc/sec air) as per ANSI N14.5. The purpose of this literature review is to supplement aging studies currently being performed by SRNL on the EPDM O-rings to provide the technical basis for extending annual re-verification testing for the H1616 shipping package and to predict the life of the seals at bounding service conditions. The available data suggest that the EPDM O-rings can retain significant mechanical properties and sealing force at or below bounding service temperatures (169 °F or 76 °C) beyond the 1 year maintenance period. Interpretation of available data suggests that a service life of at least 2 years and potentially 4-6 years may be possible at bounding temperatures. Seal lifetimes at lower, more realistic temperatures will likely be longer. Being a hydrocarbon elastomer, EPDM O-rings may exhibit an inhibition period due to the presence of antioxidants. Once antioxidants are consumed, mechanical properties and seal performance could decline at a faster rate. Testing is being performed to validate the assumptions outlined in this report and to assess the long-term performance of O-ring seals under actual service conditions.

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LIST OF ACRONYMS, TRADENAMES, AND ABBREVIATIONS

AMS	Aerospace Material Specification
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
AWE	Atomic Weapons Establishment
CSR	compression stress-relaxation
DCP	dicyclopentadiene
DiCup [®] 40C	monofunctional peroxide (Arkema)
DLO	diffusion-limited oxidation
DoD	Department of Defense
DOE	Department of Energy
ENB	ethylidene norbornene
EPDM	ethylene-propylene diene monomer
EPR	ethylene-propylene rubber (also EPM)
Flectol [®] H	1,2-dihydro-2,2,4-trimethylquinoline antioxidant (Flexsys America)
Gray	International unit of radiation absorbed dose, 1 Gray (Gy) = 100 rad
HAC	Hypothetical Accident Conditions
HX	1,4 hexadiene
ICV	Inner Containment Vessel
KAMS	K-Area Materials Storage
MS&T	Materials Science & Technology
MWD	molecular weight distribution
NCT	Normal Conditions of Transport
NNSA	National Nuclear Security Administration
Nordel [™]	DuPont-Dow Elastomers tradename for EPDM (now Dow Elastomers)
OCV	Outer Containment Vessel
RMS	Root Mean Square
Royalene [®]	EPDM tradename of Uniroyal Corporation (now Crompton Corporation)
phr	parts per hundred rubber
rad	radiation absorbed dose
SARP	Safety Analysis Report for a Package
SAE	Society of Automotive Engineers
SNL	Sandia National Laboratory
SR-350	trifunctional monomer (Sartomer)
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
T _g	glass transition temperature
TMPTMA	trimethylolpropane trimethacrylate (also TRIM)
Vistalon [™]	ExxonMobil tradename for EPDM
Zic Stick 85	zinc oxide dispersion (Rhein Chemie Corporation)

2.0 BACKGROUND

2.1 H1616 Shipping Package Design

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 [1]. The containers were initially designed and produced by Sandia National Laboratory. The containers were first certified in 1992, and 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.

Dual EPDM (ethylene propylene diene monomer) O-rings are used to seal each containment vessel. The seals are nominally compressed 25% (+/-3%) in concentric face seal grooves, with a surface finish requirement of 32 μ inch or 32 RMS. No seal lubricant is used. 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 [2]. The purpose of this literature review is to supplement aging studies currently being performed on the EPDM seals by SRNL to provide the technical basis for extending the annual re-verification testing for the H1616 shipping package and to predict the life of the EPDM seals at bounding service conditions.



Figure 1. EPDM O-Ring Location in the H1616 Container [1]

2.2 O-Ring Compound Specification

The O-rings used in the H1616 shipping package are required to meet Sandia specification SS395668. The specification dictates material composition and curing conditions that a compounder/supplier must meet. This approach differs from some shipping package designs where a commercial compound is specified (such as Parker V0835-75 in the Model 9975 shipping package) [3]. 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. Each compounding ingredient is briefly discussed below for information purposes.

The relative performance of this compound compared to other EPDM compounds is beyond the scope of this document. It is presumed that the formulation was developed for a good balance of properties. Variations in compound ingredients and the balance of such ingredients can affect both the processing and performance of the final compound and there are always tradeoffs. Changes to the formulation could occur if any of the ingredients become unavailable, but the listed ingredients are commonly used in elastomer compounding and are not unique to this specification. Nominal properties of the compound are given in Table 2.

Table 1. H1616 EPDM seal compound SS395668 [1]

<u>Ingredient</u>	<u>Parts by Weight</u>
Nordel™ 1470	100
Zic Stick 85	5
N-990 carbon black	40
N-539 carbon black	25
DiCup 40C	12
Flectol H antioxidant	2
SR-350 Sartomer	10

Nordel™ 1470: DuPont EPDM based on 1,4 hexadiene (HX), having a Mooney viscosity of 69 (ML 1+4 125 °C), average molecular weight of 290,000, broad MWD, high hexadiene level, amorphous structure, and a glass transition temperature (T_g) of -60 °C [4, 5].

Zic Stick™ 85: A very fine zinc oxide dispersion by Wyrough & Loser (later acquired by Rhein Chemie Corporation) with approximately 90% ZnO content in a proprietary binder and a specific gravity of 3.66. The product is a common activator for natural rubber and synthetic rubber, as well as a vulcanizer for polychloroprene (neoprene) and other halogenated elastomers. Superior activity coupled with rapid complete incorporation enables 3 parts of Zic Stick 85 to replace 5 parts of zinc oxide powder in most applications [6].

N-990/N-539: Carbon black is a common and important ingredient in most rubber compounds and is usually the largest volume ingredient after the base polymer. There are five main types of carbon black: acetylene black, channel black, lamp black, furnace black and thermal black. The majority of rubber grade thermal and furnace blacks are classified using a four character naming

convention per ASTM D1765 [7]. The first character is a letter that indicates the effect on the compound cure rate, with N representing a normal cure rate. Most rubber-grade carbon blacks are “N” grades. The next character is a number based on the average surface area of the carbon black (as measured by nitrogen surface area, m²/g) while the last two characters are assigned arbitrarily. The surface area range of N-990 particles is 0-10 m²/g, while the surface area range for the N-539 grade is 40-49 m²/g. The corresponding particle diameter range is 201-500 nm for the N-990 grade and 40-48 nm for the N-539 grade, respectively [7, 8]. A blend of sizes is often needed to optimize properties.

Di-Cup[®] 40C: Di-Cup[®] 40C is a monofunctional peroxide formulation of 40% dicumyl peroxide (molecular weight 270) on a CaCO₃ carrier with 2.38% active oxygen. Di-Cup[®] 40C is used for the crosslinking of many kinds of rubbers, both natural and synthetic types. It is used in many applications such as building profiles, automotive parts, wire and cable and technical rubber parts. Di-Cup[®] 40C is sold by Arkema as a free-flowing off-white powder [9].

Flectol[®] H: Flectol[®] H (1,2-dihydro-2,2,4-trimethylquinoline polymer, (C₁₂H₁₅N)_x) is a widely used general purpose antioxidant with a relatively high molecular weight and softening point. The higher molecular weight tends to promote lower migratory properties and provide superior performance with regard to staining and antioxidant persistence. The higher molecular weight also allows the material to be more easily ball-milled and evenly dispersed in rubber compounds. Flectol[®] H product is sold by Flexsys America [10, 11].

SR-350: SR-350 (trimethylolpropane trimethacrylate, also abbreviated as TRIM or TMPTMA) is a low volatility trifunctional monomer offering fast cure response in free radical polymerization, basically an efficient cross-linking coagent for improving mechanical properties. SR-350 is sold by Sartomer [12].

Table 2. Nominal Properties of H1616 O-Ring Compound SS395668 [1]

Property	Value
Hardness, Shore A	78 +/- 5
Tensile Strength, psi	1200 minimum
Elongation, percent	100 minimum

2.3 O-Ring Service Conditions

The service temperature range quoted for the H1616 seals is -40 °F (-40 °C) to 169 °F (76 °C). The NCT temperature (76 °C) is based on an ambient temperature of 100 °F (38 °C) with solar heating [1]. The maximum temperature at the flange closest to the O-rings is 152 °F (67 °C) with solar heating and 116 °F (47 °C) in the shade. The maximum temperature judged to be most applicable to the O-rings is 152 °F (67 °C). Typical seal temperatures are expected to be lower.

It is presumed that the O-rings must provide a leaktight seal as low as -40 °C, a typical transportation requirement. Since such temperatures would likely only be reached under abnormal conditions, and because the glass transition temperature (T_g) of EPDM elastomers is well below -40 °C, the EPDM O-rings will likely maintain significant resiliency after some aging. However, at some point, the seals may age enough to affect the ability to maintain a leaktight seal at -40 °C. Low-temperature performance of aged seals is not within the scope of this study.

Bounding radiation dose rates for the H1616 O-rings were not identified but are not expected to be significant relative to seal performance within the expected service time. The leak rate criteria for the EPDM seals is leaktight per ANSI N14.5, or $<10^{-7}$ ref cc/sec air. A nitrogen or argon backfill gas is specified for the H1616 containment vessels. No lubricant is used for the O-rings.

The O-rings are compressed nominally 25% in concentric grooves of rectangular cross-section, with no significant ID stretch imposed.

3.0 EPDM AGING DATA REVIEW

3.1 EPDM Chemistry, Compounding and General Properties

EPDM (ethylene-propylene diene monomer) and EPR (ethylene-propylene rubber, also EPM) polymers are synthetic elastomers developed in the 1960s, principally for aerospace and automotive applications, and in particular for resistance to phosphate ester-based hydraulic fluids such as Skydrol [11, 13]. Ethylene-propylene rubbers use the same chemical building blocks or monomers as polyethylene (PE) and polypropylene (PP) thermoplastics. These monomers are combined in a random manner to produce rubbery and stable polymers. EPDM and EPR polymers are made by halting crystallization during ethylene-propylene copolymerization, thus stabilizing an elastomeric phase well below room temperature.

EPR copolymers are amorphous and are completely saturated (single carbon-carbon bonds along the polymer backbone), with crosslinking only possible with free radicals generated by either peroxides or high-energy radiation. If a third monomer (a non-conjugated diene) is added during copolymerization, an EPDM elastomer is produced. The backbone of the EPDM elastomer remains saturated but the diene groups provide active sites for vulcanization (crosslinking) by either sulfur or peroxides or other chemical modifications. The chemical structure of EPR and EPDM polymers is shown in Figure 2.

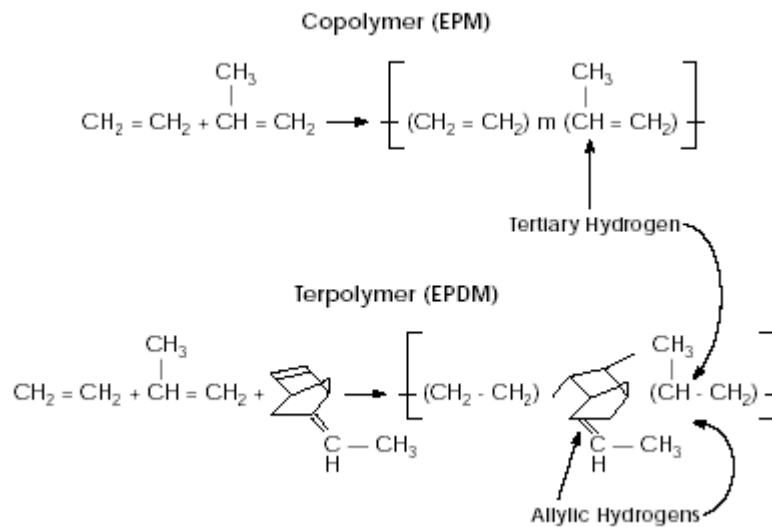


Figure 2. General chemical structure of EPR (EPM) and EPDM polymers [13]

Specialized catalysts are used to polymerize the monomers into controlled structures. Since their introduction, ethylene-propylene elastomers have used a family of catalysts referred to as Zeigler-Natta types named after their developers. More recently, a new family of metallocene-based catalysts has been developed and is in commercial use [14].

There are three major commercial processes for manufacturing ethylene-propylene rubbers: solution, slurry (suspension) and gas-phase [13]. The solution polymerization process is the most widely used. Ethylene, propylene and catalyst systems are polymerized in an excess of hydrocarbon solvent. Stabilizers and oils, if used, are added directly after polymerization. The solvent and unreacted monomers are flashed off with hot water or steam, or mechanically devolatilized. The crumb polymer is dried with dewatering in screens, mechanical presses or drying ovens, then formed into wrapped bales or extruded into pellets. High viscosity, crystalline polymer grades are typically sold in loosely compacted, friable bales or as pellets. The amorphous polymers grades are typically in solid bales.

The slurry or suspension process is a modification of bulk polymerization. The monomers and catalyst system are injected into a reactor filled with propylene. The polymerization takes place immediately, forming polymer crumbs that are not soluble in the propylene. Slurry polymerization reduces the need for solvent and solvent handling equipment, and the low viscosity of the slurry helps control temperature and product handling. The process is not limited by solution viscosity, so high molecular weight polymer can be produced.

Gas-phase polymerization technology was more recently developed for the manufacture of ethylene-propylene rubbers, eliminating the need for solvent stripping, washing and drying. The process is also not limited by solution viscosity, allowing higher molecular weight polymers to be produced.

The processing, vulcanization and physical properties of ethylene-propylene elastomers are largely controlled by the characteristics of ethylene content, diene content, molecular weight (or Mooney viscosity) and molecular weight distribution. Many grades of EPDM are available depending upon the properties required.

The three diene types most commonly used have been: dicyclopentadiene (DCP), ethylidene norbornene (ENB) and 1,4 hexadiene (HX) [11]. In the current market, the majority of EPDM compounds are based on either DCP or ENB [13]. The molecular weight range of most commercial grades of EPDM is around 200,000 - 300,000K, with a Mooney viscosity (ML 1+4 at 100 °C) range of 25-100. General features of EPDM rubber compounding are summarized in Table 3.

Table 3. General Features of Ethylene-Propylene Elastomer Compounds [13]

<u>Characteristics</u>	<u>High</u>	<u>Low</u>
Ethylene Content	Good Green Strength Flow at High Extrusion Temperatures High Tensile Strength, Modulus High Loading (Reduced Cost)	Fast Mixing Low Temperature Flexibility Low Hardness and Modulus Calendering and Milling
Diene Content	Cure Degree and Fast Rate Acceleration Versatility Good Compression Set High Modulus, Low Set	Scorch Resistance High Heat Stability Low Hardness and Modulus
Molecular Weight	Good Tensile, Tear, Modulus, Set High Loading and Oil Extension Good Green Strength Collapse Resistance	Fast Mixing High Extrusion Rates Good Calendering Low Viscosity, Scorch Resistance
MWD	Overall Good Processing Extrusion Feed and Smoothness Collapse Resistance Good Calendering and Milling	Low Die Swell Fast Extrusion Rate High Cure Good Physicals

Since the main backbone chain in EPDM is fully saturated, the elastomer exhibits excellent resistance to oxygen, ozone and many chemicals. However, additives such as antioxidants and antiozonates are generally required to maximize the weathering/aging resistance of EPDM.

EPDM is non-polar and resistant to many chemicals including most salt solutions, alkaline solutions, steam, dilute acids, acetone, alcohols and phosphate ester-based hydraulic fluids. The resistance of EPDM to organic compounds is mixed, with poor or limited resistance to aliphatic, aromatic, or chlorinated hydrocarbons or petroleum-based fluids and lubricants. It is resistant to dilute acids, but is attacked by strong acids, particularly oxidizing types such as nitric [11].

EPDM elastomer is generally resistant to temperatures of 250 °F in air, with higher temperatures tolerable with specific compounding or in certain environments or for shorter periods. EPDM is often described as a “preferred” elastomer for nuclear service due to its combined resistance to radiation, chemicals, aging, steam, heat and other environmental factors [15]. Peroxide-cured

EPDMs are generally preferred over sulfur-cured types for superior heat aging and radiation-resistance properties, but there are variations in compounding that further influence this behavior.

EPDM is sold under various tradenames such as Nordel[®] (originally DuPont, now Dow Elastomers), Vistalon[®] (ExxonMobil) and Royalene[®] (Uniroyal Chemical, now Lion Copolymer), with many individual compounds available. More recently, Dow Elastomers has been producing EPDM grades based on metallocene catalyst technology under the Nordel[®] IP tradename.

3.2 Summary of Aging Data on EPDM Elastomers

Aging of polymeric materials is a very complex subject. Many variables can influence the aging behavior, including the base polymer type, specific formulation, heat, radiation (including ultraviolet), chemicals, ozone, moisture and mechanical stress (applied and/or residual from processing). Oxidation is the primary degradation mechanism for most polymers, with fluoropolymers and silicone-based polymers being more resistant than hydrocarbon-based materials. There are also possible synergistic effects, radiation dose rate effects, diffusion-limited oxidation (DLO) effects, and many other aspects which further complicate aging behavior in multivariable environments.

The true lifetime of any material or component is the point at which the material fails to serve its intended function. For an elastomeric seal, lifetime is generally dictated by the time to leakage. The allowable leakage rate and consequences of leakage will vary with the application (liquid or gas). For many RAM packaging applications, leaktightness as defined per ANSI N14.5 (<1E-07 ref cc/sec air) is often the requirement [2]. However, in many aging studies, the time to leakage is not used to determine service life. There are several factors that can affect leakage independent of material behavior, so service life is often defined by a material property.

The most relevant property for elastomeric seals in compression is considered to be compression stress-relaxation (CSR), in absence of volume swell or other degradation due to chemical exposure [16]. Compression set (CS), which is basically the amount of dimensional recovery loss after a compressive load is removed, has historically been used as a measure of seal performance [17]. A limitation of compression set is that the value can vary with the degree and time of compression, as well as the time elapsed between load removal and measurement. Compression set is a reasonable indicator of performance, but direct comparison of results is difficult if materials do not see equivalent conditions.

In contrast, CSR is a direct measure of the counterforce imposed by the seal during actual compression. Over time, the elastomer relaxes due to physical and chemical mechanisms, such that the counterforce or sealing force is reduced. Different values of CSR (i.e. 50%, 75%, 90%) may be used to define failure, depending on the design and level of conservatism desired. Even with CSR, the minimal amount of counterforce needed in a particular configuration or design is often unknown. Under very static conditions, it may be possible that leakage criteria can be met with very low values of sealing force or even 100% loss, but this provides no margin to account for vibration, thermal changes or other mechanisms that could disturb the seal.

EPDM elastomer is widely used for many products subject to aging such as roofing membranes, cable insulation, building materials and automotive products. Therefore, many studies on the thermo-oxidative aging behavior of EPDM elastomers and the effectiveness of various compounding ingredients have been performed. However, different studies use different properties and performance parameters to evaluate degradation. Resistance to ozone, UV light and other weathering variables are important for outdoor environments, but are not critical for indoor applications. Likewise, degradation in fluid service is not generally relevant to in-air applications. Therefore, this review focused on literature judged to be most relevant to EPDM O-ring seals in the H1616 shipping package.

In many studies, ionizing radiation is a primary or secondary environmental variable (e.g. aging of EPDM or EPR nuclear cable insulation). However, as in most cases of RAM transport, the radiation doses expected for the H1616 seals are considered very low during the anticipated service period. The effects of radiation and thermal aging can be similar in some ways but significantly different in others. Therefore, studies involving significant radiation doses as part of the aging scheme were not included in this review. Relevant documents are discussed below.

SAND94-2207, "Performance Testing of Elastomeric Seal Materials under Low and High-Temperature Conditions: Final Report", D.R. Bronowski [18].

An extensive amount of testing was performed to evaluate the short-term performance of seals commonly used in radioactive material packages. This document summarizes testing that was incrementally documented in other references [19, 20].

Leak tests were performed on several O-ring compounds specified in RAM package designs. Tests were performed at high and low temperature limits, with the failure criterion at both temperature extremes being leaktight ($\leq 1E-07$ ref cc/sec air). Room temperature tests alone were not considered sufficient, presumably to address possible leakage at extreme temperatures even if room temperature performance was acceptable. At elevated temperatures, helium leak testing was complicated by rapid permeation, which can readily mask true (bypass) leakage. Therefore, the authors developed a dual tracer gas method with helium and neon for distinguishing permeation versus bypass leakage [19].

Several O-ring compounds were evaluated, including Parker Seal EPDM compounds E0740-75, E0893-80, E0540-80, as well as the V0835-75 fluoroelastomer-based compound currently used in the 9975 package and other RAM package designs. Compounds were leak tested at 380 °F (193 °C) for a 2 hour period with zero failures. Relatively low compression set values (9-13%) were measured for the short exposures. Additional tests were performed at temperatures 30 °F higher than previous tests for a 3-hour use period for a level of margin. Tests at 410 °F (210 °C) also produced no failures. No EPDM compounds were tested at temperatures > 410 °F.

Low temperature testing was also performed, with only the Viton[®] O-ring seals (V0835-75) producing leak failures at -40 °C, the low temperature rating of the material. EPDM compounds consistently showed better low-temperature performance. These results are not surprising, given the significantly lower glass transition temperature (T_g) of EPDM elastomers.

“Accelerated Aging of EPDM and Butyl Elastomers”, Federal Manufacturing & Technologies, KCP-613-5806, M.H. Wilson, 1996 [21]

The purpose of this study was to evaluate the postcure properties of a new EPDM compound developed at the University of Akron, OH and the stress-relaxation behavior of two different O-ring materials (EPDM and butyl rubber) used in weapon component designs. Accelerated compression stress relaxation tests indicated that the EPDM compound was far superior to the butyl rubber and that the optimum postcure for the EPDM was 2-4 hours at 182 °C in vacuum. The required service life for the O-rings was 20-25 years.

The butyl compound was Parker Seals B612-70 and the EPDM compound meets Sandia specification SS384725. The SS384725 compound is very similar to the H1616 O-ring specification, consisting of: Nordel[®] 1440 (100 phr), zinc oxide (5 phr), N-990 carbon black (40 phr), N-539 carbon black (25 phr), DiCup[®] 40C (12 phr), Vanox[®] ZMTI (2 phr) and Rocryl[®] 910 (10 phr). The ingredient balance is the same as the H1616 specification, but some ingredients are different, notably the Nordel[®] 1440 polymer, the Vanox[®] ZMTI antioxidant and the Rocryl[®] 910 crosslinking modifier.

The Rocryl[®] 910 crosslinking agent (ethylene glycol dimethacrylate) in the SS384725 compound is a difunctional methacrylate ester (similar to Sartomer SR206), whereas the SR-350 agent in the H1616 specification is a trifunctional methacrylate ester. Reference 21 notes that the Vanox[®] ZMTI antioxidant was later changed to Flectol[®] H to eliminate surface attack during air post-cure. The history, development and relative performance variations and requirements of the SS384725 compound and the H1616 compound (SS395668) are unknown to SRNL.

Initially, stress relaxation tests were to be performed at 60 °C, 70 °C and 80 °C for 1 year. However, early results showed no significant variation at 60 °C so those tests were dropped and tests at 70 and 80 °C were run for longer periods. Three disks (1.575 mm thick x 12.7 mm diameter) were stacked and compressed approximately 25% in CSR jigs (Shawbury-Wallace type), with three jigs used for each test temperature.

CSR results at 70 °C and 80 °C are shown in Figures 3 and 4, respectively. At 70 °C, the EPDM compound maintained a sealing force ratio of ~ 0.70 after 616 days (1.7 years), whereas the sealing force ratio of the butyl compound dropped from ~ 0.55 to around 0.10 (Figure 3). At 80 °C (Figure 4), the sealing force ratio of the EPDM compound dropped only slightly more (~0.65) in 616 days, while the butyl rubber ratio dropped to around 0.35 in ~100 days.

These test temperatures are notably similar to the NCT temperature (76 °C) and the bounding O-ring temperature (67 °C) anticipated for the H1616 O-rings. Given the similarity in compound formulation, these data strongly suggest that the H1616 compound will retain significant properties and sealing force for at least 1.7 years at these temperatures and possibly well beyond as these are not actual failure times. In the absence of immediate rapid oxidation and corresponding loss in properties, these data alone suggest that a service period of at least 2 years is reasonable at these temperatures. Longer life is expected at lower temperatures.

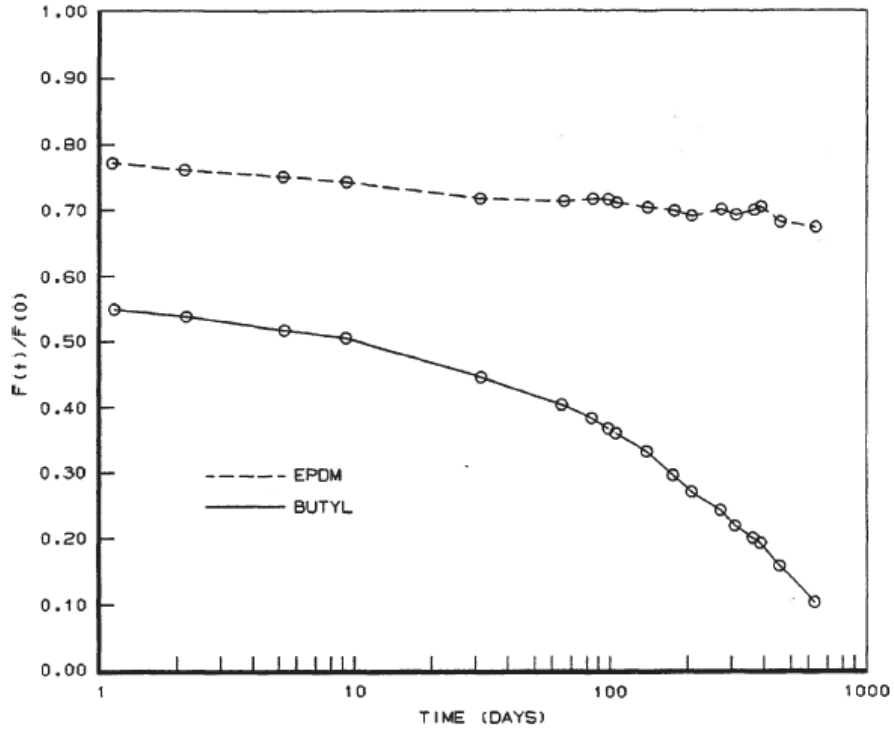


Figure 3. Stress Relaxation of Butyl and EPDM Elastomers at 70 °C (616 days) [21]

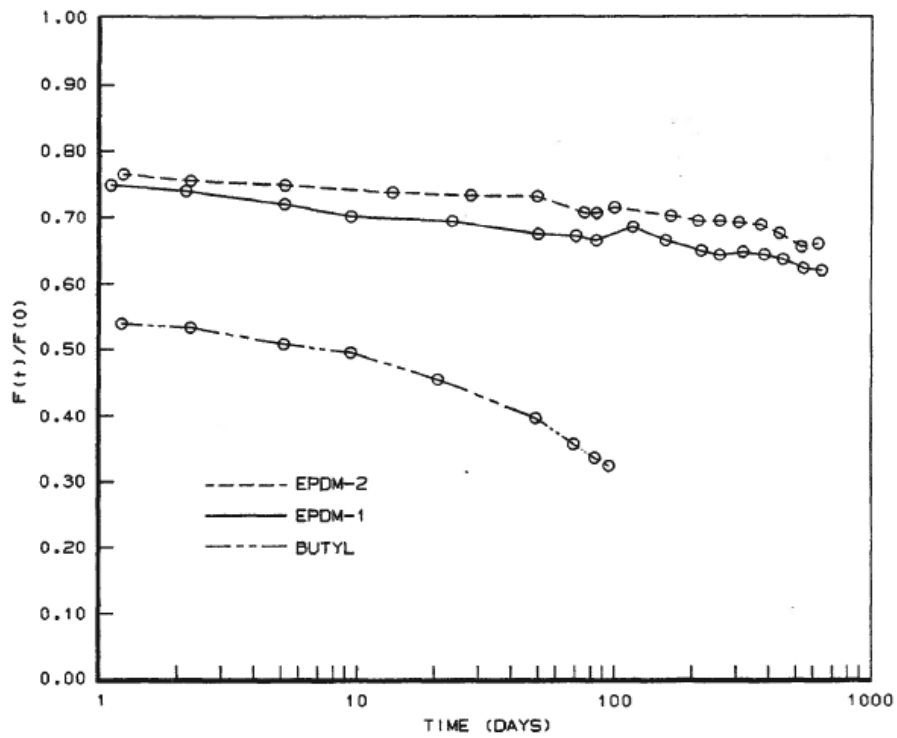


Figure 4. Stress Relaxation of Butyl and EPDM Elastomers at 80 °C [21]

SAND98-1942, "New Methods for Predicting Lifetimes in Weapons, Part 1: Ultrasensitive Oxygen Consumption Measurements to Predict the Lifetime of EPDM O-Rings", K.T. Gillen, M. Celina, R.L. Clough, G.M. Malone, M. Keenan, J. Wise [22]

In Reference 22, investigators evaluated the lifetime of EPDM O-rings for weapon components at elevated temperatures to predict service life at ambient temperature (25 °C). EPDM O-rings were aged at temperatures of 110 °C to 155 °C. Various properties including elongation, density, modulus and compression stress-relaxation behavior were examined. The EPDM compound evaluated in this study (SR793B-80) was based on Nordel[®] 1440. This compound is notably similar to the H1616 compound, with the only variation being the base polymer (Nordel[®] 1470 in the H1616 specification). The reason for the base polymer variation between these compounds is unknown to SRNL. 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 the 1470 grade having a Mooney viscosity of ~69 and a molecular weight of ~290,000 [4, 5].

Conventional extrapolation of aging data from higher temperatures to 25 °C yielded unrealistically long lifetimes (55,000 years). The oxygen consumption rate of the elastomer was subsequently evaluated at temperatures down to 52 °C to determine the possibility of non-Arrhenius behavior due to a shift in the degradation mechanism. The activation energy (E_a) was noted to drop by ~30% to 82 kJ/mol below 110 °C, significantly reducing the predicted lifetime to ~150 years at 52 °C and ~2000 years at 25 °C (Figure 5).

The purpose of the work performed in Reference 22 was to predict seal performance at room temperature. However, the advantage of time-temperature superposition is that the data can be translated to any temperature of interest, assuming a constant activation energy. The CSR data showed that a ~90% loss in sealing force ($F/F_0 < 0.1$) was observed after ~200 days at 125 °C and after ~580 days (1.6 years) at 111 °C. These data are superposed in Figure 6. These are not absolute limits in terms of seal function, but significant degradation is indicated. Note the 111 °C temperature is very close to the 113 °C aging temperature currently being used in the SRNL study on the H1616 O-rings. So it may be reasonable to expect similar behavior within the same period of time at this temperature.

Changes in surface modulus, density and elongation properties were also measured, as such properties have been shown to be less sensitive to cross-section thickness and the possibility of diffusion-limited oxidation (DLO) effects. The authors suggest from these tests that the EPDM may show an "induction" period related to antioxidant protection or other mechanisms. Beyond this period, changes in properties are more rapid.

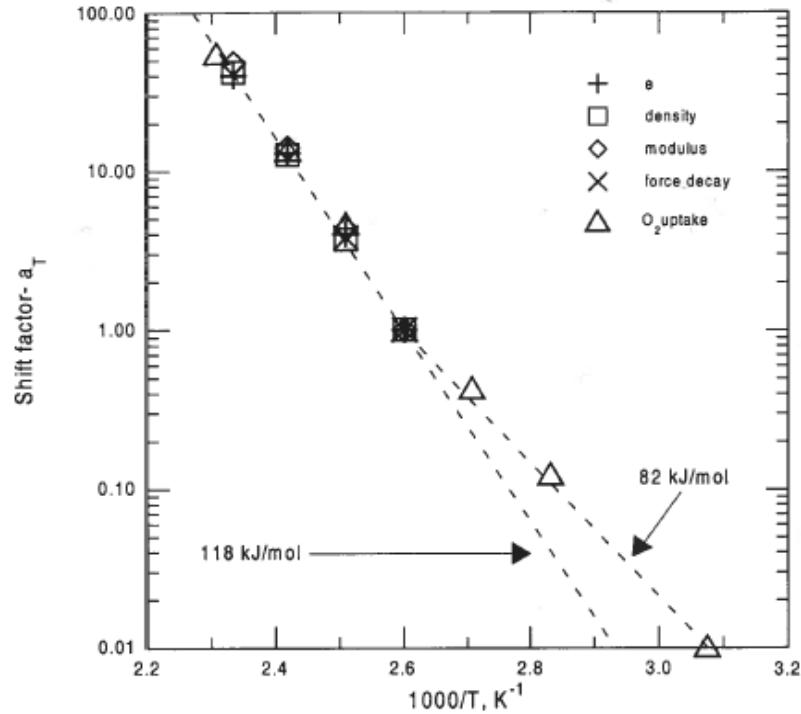


Figure 5. Arrhenius plot of shift factors for EPDM vs. inverse absolute temperature [22]. (oxygen uptake data shows ~30% reduction in the activation energy to 82 kJ/mol below 110 °C)

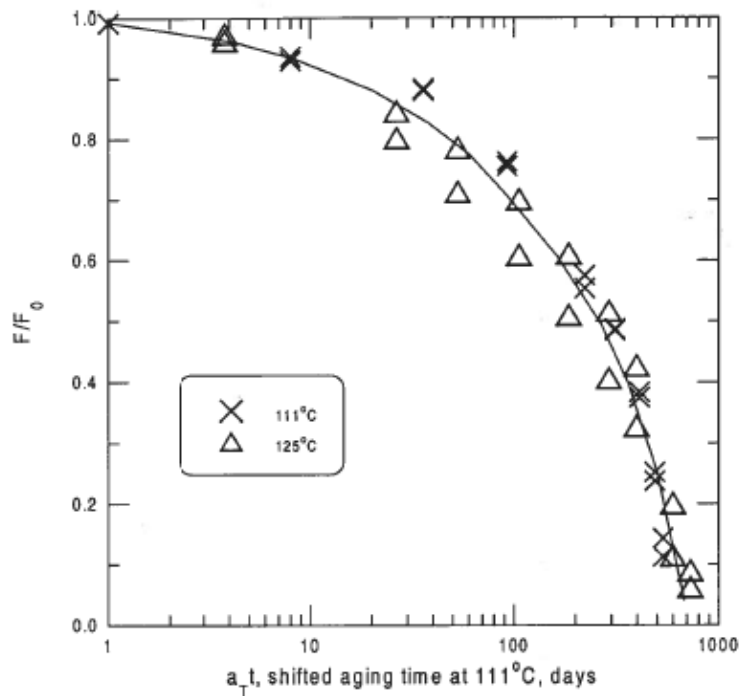


Figure 6. Time-temperature superposition of CSR data for EPDM (SR793B-80 based on Nordel® 1440) at a reference temperature of 111 °C [22]

From Figure 5, the shift factors for any temperature can be determined and used to translate the sealing force data in Figure 6 to any temperature of interest. For example, the 150 year service life quoted by the authors at 52 °C (325K, $1/T = 0.00308$) is derived from the time to failure (defined as $F/F_0 = 0.10$) at 111 °C (~580 days) divided by a shift factor of ~ 0.0103 estimated from the activation energy plot of 82 kJ/mol on Figure 5, or $580 / 0.0103$ days ~ 155 years.

Using this approach, a shift factor of ~0.035 is estimated for the bounding H1616 O-ring temperature of 67 °C (340K, $1/T = 0.00294$), reducing the lifetime to $580/0.035$ days or ~ 45 years. Similarly, a shift factor of ~0.08 at the NCT temperature of 76 °C (349K, $1/T=0.00287$) reduces the seal lifetime to $580/0.08$ days or approximately 19 years.

Though an excellent reference, a limitation is that the data were for a different EPDM compound (different base polymer) and the CSR tests were only performed at two temperatures for a maximum aging time of 1.6 years. The activation energy for relaxation in the H1616 O-rings could possibly fall to lower values at lower temperatures (particularly if not related to oxidation). However, these data provide a good estimate of H1616 O-ring performance at similar temperatures and provide activation energy values for comparison.

SAND97-2181C, "Evidence that Arrhenius High-Temperature Aging Behavior for an EPDM O-ring Does Not Extrapolate to Lower Temperature", K.T. Gillen, J. Wise, M. Celina, R.L. Clough [23]

This report documents some of the early work outlined in *SAND98-1942*, previously discussed. EPDM formulation SR793B-80 was subjected to conventional oven-aging experiments and measurements of tensile elongation, density and modulus properties. Compression stress relaxation (CSR) measurements were made at 125 °C and 111 °C. Oxygen consumption measurements were made over time at temperatures ranging from 160 °C to 52 °C. Using conventional Arrhenius methodology, extremely long lifetimes (55,000 years) were predicted for the EPDM O-ring compound at 25 °C. However, by using an ultrasensitive oxygen consumption analytical technique, activation energy values were found to change in the extrapolation region by 30% (116 to 82 kJ/mol) below 110 °C. This indicated that a change in the degradation mechanism occurred was possible at lower temperatures. This improved methodology resulted in a predicted lifetime of ~150 years at 52°C and ~2000 years at 25 °C.

SAND2000-0715, New Methods for Predicting Lifetimes – Part 2: The Wear-Out Approach for Predicting the Remaining Lifetime of Materials, K.T. Gillen, M. Celina [24]

An extension of work presented in Reference 22, researchers incorporated the “wear-out” approach for predicting the lifetime of several different elastomers. The wear-out approach is based on the Palmgren-Miner concept that degradation of materials is cumulative (Miner Rule) and that failure is the direct result of accumulation of damage with time. The “wear-out” approach was applied to the data obtained for the SR793B-80 compound in Part 1 of this program as well as to field-aged EPDM O-rings in ambient weapon environments [25]. In this case, the "lifetime" of the O-rings was defined as the time for the ultimate elongation of the material to reach 50% absolute (or ~10% of initial).

EPDM O-rings (Parker Seals E529-60) were examined from a retired weapon that had been aged

for 23 years in the field. Fortunately the same compound was still produced (no formulation changes noted), so new O-rings were obtained and used for comparison. Since earlier work showed that the point at which significant loss in sealing force occurred was well correlated with significant changes in other properties (elongation, density, modulus), elongation was selected as the parameter for evaluation.

140 °C (284 °F) was selected as T_w (wear-out temperature) to yield convenient wear-out times (several months). As shown in Figure 7, an absolute elongation value of 40% (10% of initial) was reached after ~70 days at 140 °C. Significant changes in density were also indicated after similar aging (Figure 8). Only density measurements were made on the 23-year old material due to limited quantity. With very little density change between aged and non-aged material, it was concluded that very little degradation had occurred in the surveillance material after 23 years at ambient weapon conditions, consistent with previous predictions. Assuming a “failure” density of 1.24 g/cc (corresponding to a 90% loss in elongation), the wear-out approach estimated a lifetime of ~320 years at ambient conditions.

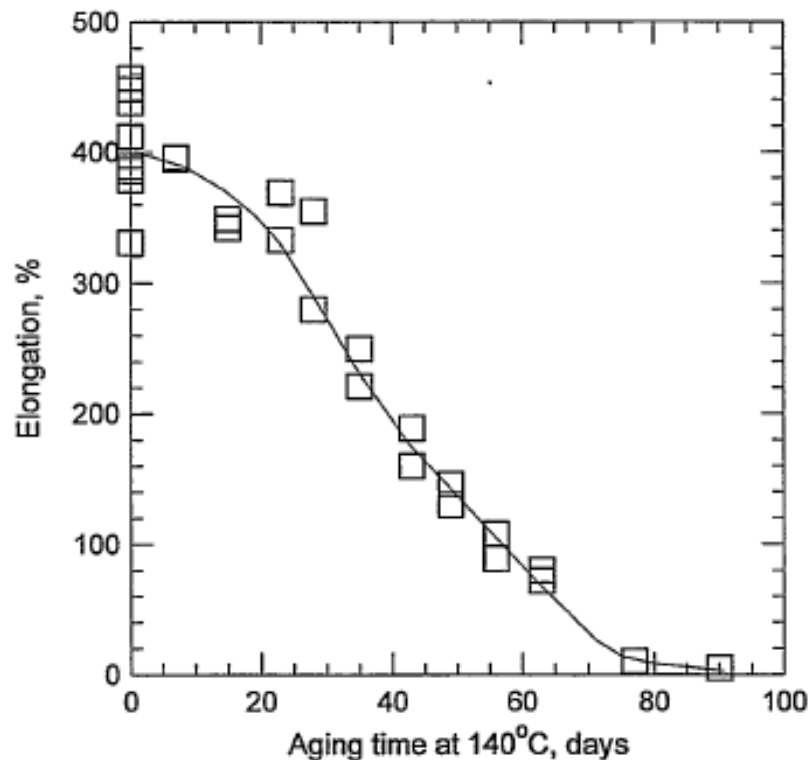


Figure 7. Elongation versus aging time at 140 °C (Parker E529-60 EPDM O-rings) [24]

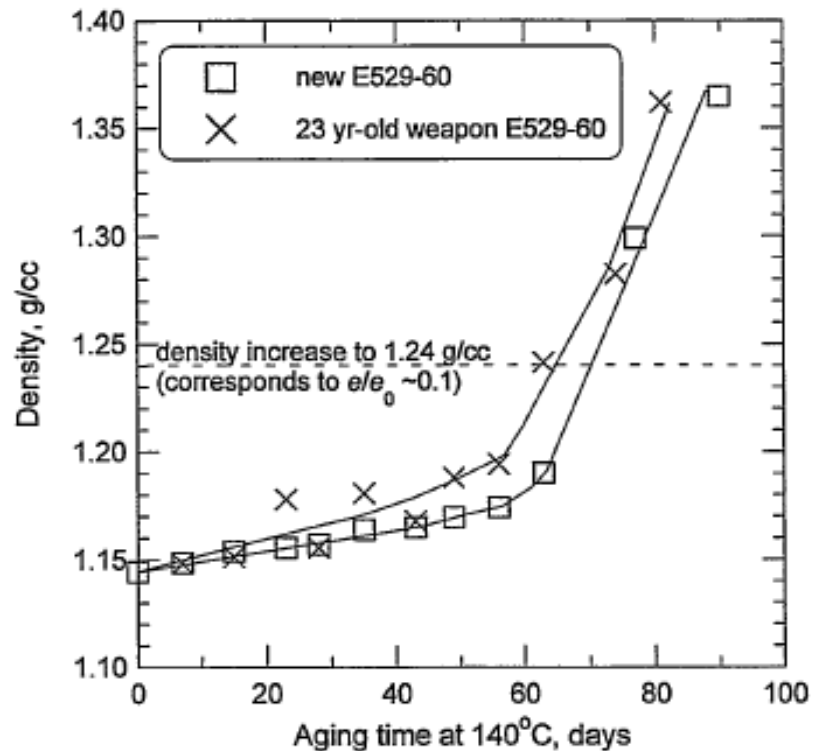


Figure 8. Density versus aging time at 140 °C (new EPDM O-rings vs. field-aged O-rings) [24]

SAND00-8259, “Experimental Evaluations of O-Ring Thickness Variability and Long-Term Compression Set and Compression Set Recovery”, L.A. Domeier [26]

In this study, several different O-ring materials used in weapon systems were evaluated for thickness variability and compression set/recovery behavior after being compressed for one year at temperatures of 60, 70, and 80 °C. Of particular interest was butyl rubber, widely used in weapon systems due to low permeability. An EPDM compound (Parker E692-75), a silicone compound, and a Viton[®] compound (V894-90) were also included.

Half-ring samples were compressed 25%. Viton[®] and silicone O-rings showed the largest initial thickness deviation but also the lowest compression set at the temperatures evaluated, consistent with higher heat resistance. The nitrile compound (Parker N756-75) showed the poorest aging performance, with butyl also degrading more than EPDM. The Viton[®] and silicone compounds both exhibited less change in compression set behavior over the one year period, with the silicone showing a higher rate of increase in compression set than the Viton[®] compound of much higher hardness (90A). Only the nitrile and butyl compounds showed compression set values >30% over the one year period at all three temperatures. At one year, the average compression set for the nitrile compound was ~95% at 80 °C and 61% at 60 °C, compared to 33% and 23% for EPDM and only 15 and 13% for Viton[®], respectively.

Time-temperature superposition techniques were used to estimate compression set at 60 °C for longer periods. Shift factors were not linear with temperature and no Arrhenius extrapolations to ambient conditions were attempted. It was estimated that the nitrile O-rings would reach 90% compression set after 1000 days at 60 °C while the butyl compound would take 4000 days or 11 years to reach the same value. Behavior of the EPDM was estimated to be similar if not superior to that of the butyl rubber, particularly at 80 °C. The relationship between compression set and leaktight performance in the H1616 application has not been established, but these data suggest that the EPDM compound could last for several years at 80 °C.

“Seal Life of EPDM O-Rings At High Temperature Determined by Unique Method”, R. Marlier, R. Andre, P. Malesys, H. Issard, Packaging, Transport Storage & Security of Radioactive Materials, Vol.17, No.1, 57-62, 2006 [27]

Researchers investigated the service life of EPDM elastomers used in RAM packaging designs, specifically the time to leakage at elevated temperature. This reference is one of few published documents to emphatically note that the “continuous” service temperatures of elastomers quoted by suppliers and seal manufacturers are often generic and based on limited data (typically 1000 hours). The authors also noted that while many publications deal with the evolution of the mechanical characteristics (i.e. stress-relaxation) of the elastomers at high temperature over time, the real parameter of interest is time to leak failure.

The investigators developed a leak test assembly and method for measuring leak rate periodically on O-rings during the aging scheme. Two test rigs were made up of two flat flanges (no grooves) separated by a spacer to adjust the degree of compression, which for these tests was approximately 30%. The O-rings tested had nominal dimensions of 190 x 10 mm.

Initial tests were carried out at 250 °C for 2 weeks, with the leak rate measured daily. After 2 weeks at 250 °C, the O-rings tested were still leak tight. The basis for testing at 250 °C was not provided, but this temperature is well above the normal service temperature for EPDM elastomer. Upon disassembly, the rigs were difficult to take apart because of sticking between the O-rings and the flanges. The O-rings were very damaged (no elastic recovery, completely vitrified). The O-rings were physically leaktight, but not due to preservation of good mechanical characteristics.

The procedure and the test assembly were modified to avoid sticking of the O-rings. The improved procedure involved separating/reassembling the flanges before each leak test. The test assembly was modified to enable the flanges to be separated. Elastic washers were added between the flanges of the two test rigs and a system of pneumatic cushions (placed outside the oven) was used to separate and to tighten the flanges. Separation and reassembling of the flanges is done automatically just before the leak test.

EPDM O-rings were aged at temperatures of 190, 200 and 210 °C, which are all high temperatures relative to typical service limits for EPDM elastomers (120-149 °C). Based on the time to leakage failure at these temperatures (2 days at 210 °C, 2 weeks at 200 °C and 2 months at 190 °C), the authors developed an Arrhenius aging model and extrapolated service life values of 2 years at 170 °C and 20 years at 160 °C. A cumulative damage model was also proposed

using a method similar to that used in fatigue studies (Wöhler curves). This approach theoretically allows the package designer to predict long-term aging effects at various bounding normal temperatures plus accounting for a shorter excursion during accident conditions.

The approach of using the time to leakage as the performance parameter has merit and the modified test apparatus and leak test methodology offers advantages in preventing false positive results (leaktight due to sticking). However, the leak test data presented are limited to 2 months or less duration and Arrhenius predictions from such data are tenuous. A further limitation of the Arrhenius approach is that only a few time to failure data points are used.

“First Test Results for Determination of Seal Life of EPDM O-Rings at High Temperature (determined by unique method)”, R. Marlier, Packaging, Transport, Storage & Security of Radioactive Materials, VOL 21 NO 1, 2010 [28]

As a follow-up from Reference 27, additional test data and aging models were reported for EPDM O-rings. The method primarily involved measuring the leak rate of O-rings fitted on test rigs every day until the leak rate criterion was reached. A 30 second helium injection period at high temperature was used to show if a bypass leak would occur, as indicated by a rapid increase of the spectrometer signal when helium is injected on one side of the O-ring and a discontinuity in the spectrometer signal when the injection is stopped.

From these results, a seal life aging model was developed. Test temperatures were 200 °C, 210 °C, 225 °C and 230 °C, all noted to be well above the typical service temperature limit of EPDM elastomers. The basis for testing at these higher temperatures was not provided, but possibly to accelerate failure within a reasonable test period.

It was concluded that new degradation mechanisms started at 230 °C (thermal activation), which drastically reduces the seal life of O-rings. Therefore, 230 °C was considered the upper limit of the law validity range for the aging model. Leak test results showed a seal life of 24 to 296 hours at 230 °C, 1236 hours at 225 °C, 1579 hours at 220 °C, 3330 hours at 210 °C, and up to 6000 hours (250 days) at 200 °C. Using these data, the authors developed an Arrhenius aging model. The model predicts a seal life of at least 100,000 hours (4167 days or 11.4 years) at 149 °C (300 °F). Projections for lower temperatures were not shown.

While the leaktight performance of EPDM seals at the temperatures tested without sticking is impressive, the aging period is still relatively limited for long-term life prediction at lower, more realistic service temperatures. The test temperatures are well above the typical service temperatures for EPDM elastomers (≤ 149 °C) and the service conditions expected in the H1616 shipping package. The data are limited to 250 days and represent a limited number of fixtures. Leak test methods and interpretation of the data can vary between investigators. These data also do not account for the possibility of non-Arrhenius aging behavior at lower temperatures over longer aging periods. If valid for lower temperatures, the model suggests that EPDM O-rings in the H1616 package should last at least 10 years at temperatures below 149 °C. However, testing at lower temperatures for longer aging periods is needed to validate the model.

“Effects of Some Antioxidants on the Stability of Ethylene-Propylene Elastomers”, T. Zaharescu, M. Giurginca, I. Mihalcea, Polymer Degradation and Stability 50 (1995) 151-154 [29]

The effectiveness of various antioxidants used in EPDM compounds were studied via infrared spectroscopy and oxygen uptake measurements. Of the antioxidants evaluated (phenols, bisphenols, this-bis-phenols, propionic and triazine compounds), the bisphenol with methylenic bridge provided the best protection for all test conditions. At temperatures below 180 °C, phenols are transformed to quinines which continue to prevent oxidation during post-induction stages. All tested antioxidants provided long-term protection compared to uncompounded elastomers or those containing weak antioxidant (Topanol OC).

“Stabilisation of EPDM at High Temperatures by Polymer-Bound Antioxidants: Part I – Air Oven Ageing: Stress Relaxation and Mechanical Property Change”, G. Scott, S.M. Tavakoli, Polymer Degradation and Stability 19 (1987) 29-41 [30]

EPDM compounds modified with various antioxidants were aged in an air oven at 150 °C, and evaluated by stress relaxation tests and physical property changes after aging at 180 °C. The EPDM polymer used in this study was Vistalon[®] 6505, with a Mooney viscosity of 80 (at 110 °C) and 50 (at 127 °C). The Vistalon[®] 6505 polymer is based on ethylene norbornene (ENB) diene, rather than 1,4 HX as in the case of the H1616 O-ring compound. The antioxidants evaluated in this study included 4-mercaptoacetamidodiphenylamine (MADA, I), 4-mercaptopropionamido diphenylamine (MPDA, II), and a commercially available antioxidant (Flectol[®] H) in both 1% and 2% concentrations. The primary purpose for the study was to evaluate the stabilization of EPDM against chemical and physical change under a variety of thermal oxidative conditions.

Continuous stress-relaxation tests were performed using a Wallace stress relaxometer with each formulation being aged at 150 °C or 180 °C in an individual cell with an air flow of 1 ft³/hr. Mechanical property changes were also evaluated using dumbbell test pieces aged in individual cells (Wallace oven) at 120 °C and 150 °C with the same air flow rate. The data shows that the MADA antioxidant slightly improves stress-relaxation behavior compared to Flectol[®] H over a 50 hour period at 150 °C. However, both the MADA and Flectol[®] H antioxidants show superior behavior to the control sample, a carbon black loaded sulfur-accelerated vulcanizate with no antioxidant. The control sample reached a 50% loss in sealing force (relaxation) within 40-45 hrs, while samples with both MADA and Flectol[®] H antioxidants lost only 20% and 30% of initial sealing force values within 50 hours, respectively.

All of the arylamine antioxidants, including Flectol[®] H, also reduce the level of cure of the sulfur accelerator vulcanizates and the scorch time. The conclusion of this study was that polymer bound antioxidants (MADA and MPDA) based on thiol adduct formation (product of a direct addition of two or more distinct molecules, resulting in a single reaction product containing all atoms of all components) in EPDM show better performance in oven aging tests than low molecular weight and oligomeric (short-length chain) antioxidants (such as Flectol[®] H). The performance of MADA at 1% is better than at 2% and extraction of the rubber adduct before compounding improves this trend.

“Stabilisation of EPDM at High Temperatures by Polymer-bound Antioxidants: Part 2 – Mechanical Property Change after Extraction During Compression Relaxation in Air”, G. Scott, S.M. Tavakoli, Polymer Degradation and Stability 19 (1987) 43-50 [31]

Related to Reference 30, fluid immersion tests were performed on EPDM compounds containing bound antioxidants MADA-B (bound). Skydrol LD-4 aircraft hydraulic fluid was used for the immersion tests. Samples were placed in bottles and closed to the atmosphere, heated to 100 °C and removed at weekly intervals with fluid renewed. Mechanical properties were measured. Additional samples were tested in a cyclic immersion-oven aging scheme, with each cycle consisting of immersion in Skydrol at room temperature for 6 hours followed by air oven aging and mechanical property measurement every 48 hours. The total aging time was 4 weeks at 120 °C. Sealing force measurements were also carried out on O-rings using a Lucas CSR instrument.

Tests again showed that the polymer bound antioxidant was superior to the conventional oligomeric antioxidant (2% Flectol[®] H) and that 1% of the polymer bound antioxidant is more effective than 2% in inhibiting oxidative change. It is important to note that though the polymer-bound antioxidants did show superior behavior at the test temperatures involved, the relative property values for all samples were not largely different.

For example, in stress relaxation tests, the percent change in sealing force for sulfur accelerator-cured samples with polymer-bound antioxidant ranged from 51-60% after 72 hours at 150 °C (with carbon black), compared to a 66% change in samples with Flectol[®] H antioxidant aged at the same conditions. Control samples (no antioxidant) showed 83% drop in sealing force at the same conditions. Interestingly, the changes in sealing force for samples aged at 150 °C were not significantly different than for samples aged at 22 °C for 72 hours.

For sulfur-peroxide cured EPDM samples, the change in sealing force for carbon-black filled samples with MADA-B antioxidant, aged at 150 °C for 72 hours, ranged from 44 to 57% (with both 1 and 2% antioxidant). In comparison, the sealing force for similarly cured samples dropped 67% for samples with 2% Flectol[®] H and 80% for the control sample.

The authors noted several observations, including that compression stress-relaxation tests in air ovens are less discriminating for antioxidant performance than extension stress-relaxation tests under similar time/temperature aging conditions. This is also likely related to the variation in sample thickness as O-ring samples used in compression tests were thicker than samples used in tensile relaxation tests. This is reasonable as oxidation is a diffusion-limited process.

3.3 Relevance of Literature Data to the H1616 O-Rings

The relevance of literature data to the H1616 O-rings varies with individual studies and methods employed. The use of mechanical parameters such as elongation, density, modulus and compression stress-relaxation to evaluate seal aging performance is common. Leakage rate is often the true measure of performance but fewer studies use this parameter. One reason for this is that leakage rate, even at very static conditions, can depend on design parameters as well as mechanical behavior of the seal material.

Some studies have shown that leaktight seals can be maintained at very low values of sealing force (~ 1 N/cm) or at 100% compression set but these values are not conservative and cannot be considered universally true for all designs [32, 33]. Oxygen consumption studies have been shown to be helpful in identifying non-Arrhenius aging behavior at lower temperatures than predicted by CSR measurements. Therefore, relevant oxygen consumption data for EPDM will be used to extrapolate the SRNL aging data for EPDM O-ring performance in the H1616 shipping package. Additional oxygen consumption testing of the H1616 compound may be beneficial to further refine such predictions for long service periods, however, the current scope of SRNL aging tests does not include oxygen consumption analysis.

The use of leak testing at highly elevated temperatures relative to service can provide an estimate of seal lifetime at lower temperatures. However, leak failures must be observed at reasonable aging temperatures to predict failure at relevant service temperatures and selection of the aging temperatures is critical to avoid misleading results (too aggressive, different mechanisms, leaktight due to sticking, etc.). Extrapolations of short term results from excessively high aging temperatures to lower temperatures, regardless of the parameter selected, are more tenuous.

A few of the reviewed references appear to be the most relevant to the H1616 shipping package (References 21, 22, 24, 27, 28). Data in Reference 21 suggests that mechanical properties and sealing performance will be highly retained at the bounding temperatures (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.

Translation of time-temperature superposed CSR data from Reference 22 suggests possible lifetimes of ~ 150 years at 52 °C, ~ 45 years at 67 °C (max O-ring temp with solar heating) and ~ 19 years at 76 °C (NCT temperature), assuming an activation energy of 82 kJ/mol and failure defined as 90% loss in sealing force. Given that the O-rings in the H1616 shipping package may see lower temperatures due to reduced payload and/or reduced ambient temperatures, the H1616 seals may be able to function for even longer periods. These data are for a very similar EPDM compound, though with a different base polymer, and the data are limited to two temperatures (111 °C, 125 °C).

Data from References 27 and 28 suggest EPDM O-rings can maintain leaktight seals for at least 10 years at temperatures below 149 °C. However, such tests do not address the potential for non-Arrhenius aging behavior over longer aging periods at lower temperatures. Longer aging periods at lower temperatures are needed to validate the aging models presented in these references

A summary of the EPDM seal lifetimes observed or predicted at various temperatures from different references is given in Figures 9 and 10. Direct comparison is difficult as the basis for performance or failure in each reference varies. All of the lifetime data are given in Figure 9, with a narrower temperature range shown in Figure 10 for clarity. Data points represented by solid symbols (References 18 and 21) are not actual failures as defined by CSR or leakage.

The trendlines shown in Figures 9 and 10 are intended to show the most reasonable lifetimes that can be predicted from the available data. Actual lifetimes could be longer. At 76 °C, the trendline suggests a lifetime of approximately 4.2 years. At 67 °C, the trendline suggests a lifetime of ~ 5.8 years. At 47 °C (max O-ring temp in shade), the lifetime is increased to about 19 years.

Service life of the seals is likely highly dependent on actual temperatures. For lower temperatures, the lifetime is accordingly increased. It is important to note that these predictions do not inherently mean that the O-rings will cease to provide a leaktight seal at these time periods. Leaktight performance may be achievable well beyond periods defined by CSR failure.

Such predictions also assume that the behavior of the H1616 compound will mimic that of the compounds evaluated in other studies. Given that at least some of the data reviewed are for similar compounds involving the same antioxidant and amount (Flectol[®] H), this assumption is likely valid. The relative aging performance of the H1616 compound based on Nordel 1470 is unknown. The higher molecular weight of the 1470 grade will possibly improve properties such as stress-relaxation. However, predictions can only be validated by longer tests at lower, more realistic temperatures.

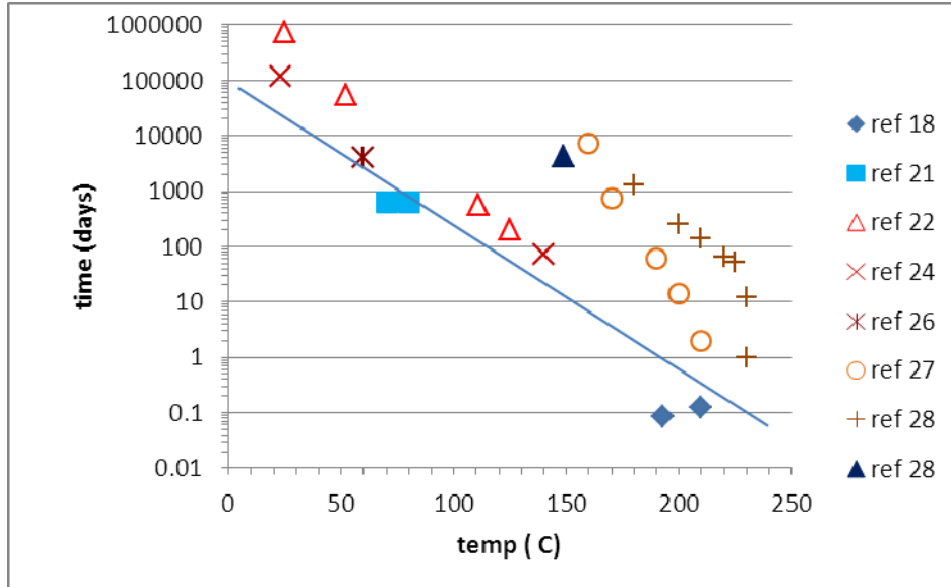


Figure 9. EPDM O-Ring Lifetime Data from Literature References

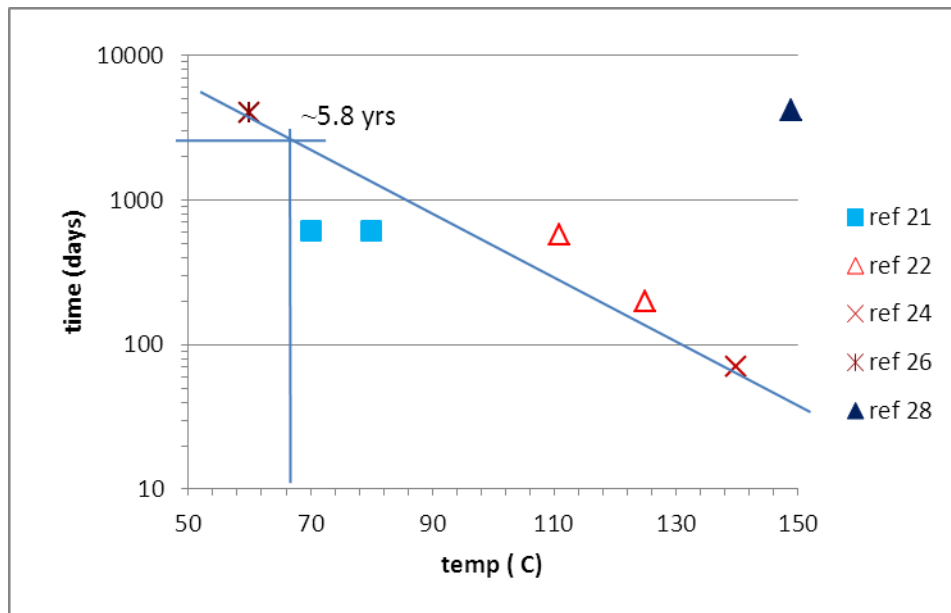


Figure 10. EPDM O-Ring Lifetime Data (narrower temperature range)

4.0 CONCLUSIONS

- 4.1 Collective literature data suggest that the H1616 EPDM O-rings can likely retain sufficient properties to remain leaktight for at least two years at or below 76 °C, the NCT temperature. Interpretation of accelerated-aging data from various references suggests possible lifetimes of at least 4 years at 76 °C and at least 6 years at 67 °C (peak O-ring temperature with solar heating). Lifetimes should be longer at lower service temperatures. Leaktight performance may be achieved longer than indicated by CSR data. Aging tests are currently in process to validate these interpretations.
- 4.2 The EPDM O-rings may show an “induction” or inhibition period due to antioxidant protection. Beyond this period, mechanical properties and sealing force may show more rapid degradation. The antioxidant (Flectol[®] H) used in the H1616 compound is the same as used in some of the compounds evaluated in the data review. The inhibition period in actual service will depend on several variables including antioxidant stability, oxygen access and compound-specific behavior. Depending upon the results of the SRNL aging study, oxygen consumption analysis may be beneficial to further refine aging model predictions for long service periods. However, the current scope of SRNL aging tests of the H1616 EPDM compound does not include oxygen consumption analysis.
- 4.3 There is no substitute for real-time aging at realistic or bounding conditions, as accelerated aging methods have inherent limitations. Periodic examination or testing of actual seals from a select group of H1616 shipping packages aged at bounding conditions may be useful to validate assumptions and predictions made 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.
- 4.4 The performance of aged seals at HAC conditions (post-fire or low temperature) is not part of the current SRNL study. With a low glass transition temperature (-50 °C or lower), EPDM seals have significant margin at -40 °C such that performance after some aging is likely acceptable. However, at some point, aging may be significant enough to affect seal performance at such conditions. Testing of seals at extreme conditions after aging may be necessary to establish acceptable limits.
- 4.5 One aspect of the H1616 O-ring application and other RAM packages that is difficult to assess is the potential effect of multiple use cycles during the maintenance period. Multiple closure cycles may have both positive and negative effects. Periodic release of the compressive load may provide some elastic recovery, but the cumulative relaxation effect may be more severe than in a static case. Multiple use cycles may also accelerate antioxidant depletion, though the gas backfill (N₂ or Ar) may reduce this effect.

5.0 ACKNOWLEDGMENTS

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