Elastomer Service Life Prediction in Organic Acid Coolants

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ABSTRACT

The objective of this test program was to review the potential physical and chemical changes of four candidate elastomers in engine coolant service. The glycol-carboxylic acid inhibitor technology coolant was selected due to its frequent use and appearance of being more environmentally acceptable as compared to currently used inorganic inhibitors. Within the limits of the test program, we reviewed physical aging using oring seals (ASTM D1414) and compressive stress relaxation. The chemical changes were monitored by reviewing surface changes (Fourier Transform Infrared Spectroscopy, FTIR) using attenuated total reflectance (ATR) and dynamic mechanical and rheological testing/thermal analysis, DMRT, which should reflect bulk property changes due to chemical attack.

INTRODUCTION

Testing to determine suitability for long-term sealing service should incorporate measurement of data meaningful to the application as well as apparatus that is a legitimate model of the application. Long-term seal performance prerequisites might logically start with the elastomer having a minimal potential for chemical change and/or long-term resistance to compression set and compressive stress relaxation. The test program and apparatus used for this paper were designed to provide data relevant to elastomer selection for engine coolant applications.

Premature failure can occur due to chemical attack on the base elastomer (a.k.a. backbone), the crosslink, or both. Physical relaxation (stress/strain relaxation) can lead to premature failure of seal contact stress and is typically accelerated by thermal excursions (Boltzman Superposition Principle). The chemical attack of polymers in organic acid coolant is well documented (1) (2). The initial chemical changes on the elastomer's surface can be monitored via Fourier Transform Infrared

Spectroscopy (FT-IR) with the Attenuated Total Reflectance (ATR) technique.

Chemical stability of base monomers largely determines Crosslinking chemistry is likewise polymer stability. predictable via named (e.g. Swarts, Ene, etc) chemical reactions. The bulk chemical changes are logically timetemperature dependent; these changes can be monitored by dynamic mechanical and rheological thermal analysis (DMRT). DMRT testing is performed under shear (a linear elastic response). Shear modulus, G, of an elastomer is generally expressed as G = $\rho RT/M_c,$ where ρ is density, R the universal gas constant, and T is temperature in Kelvin. controversial term, is molecular weight between crosslinks. Considering Mc as the variable reflecting chemical changes that occur during the aging process we can now measure the elastomer's bulk properties as they shift due to aging.

Maintenance of contact stress between elastomeric elements and mating surfaces is critical to proper sealing. Measurement of compressive stress relaxation, or CSR, offers valuable insight for prediction of long-term suitability of materials. A specimen geometry that reflects a typical seal cross section and realistic operating temperature were used in this papers test program to accurately model environmental conditions of seals in coolant service.

EXPERIMENTAL

OBJECTIVES

The authors wished to compare and evaluate four different elastomers with respect to long-term sealing performance in aggressive service environments-particularly high temperature service in glycol/carboxylic acid inhibited coolant. A popular coolant fluid was used to test chemical resistance and thermal stability. A test fixture was designed to closely simulate the conditions of various engine cooling components. For a material to

have long service life, it should withstand both dry heat aging as well as submersion in the coolant fluid.

In a previous paper (1) the authors used more aggressive aging temperatures (150, 175 and 200°C) with EPDM, FEPM, and HNBR with both carboxylic and inorganic inhibited glycol coolants. The inclusion of a silicone (VSi) elastomer in this paper dictated testing at a lower temperature, namely 135°C. This is equivalent to a bulk coolant temperature of 100°C with a heat rejecting source at 135°C. This aging temperature was within the recommended service temperature limits of each elastomer.

Ideally, the elastic properties of an elastomeric material would remain unchanged after any amount of time in service. Obviously, exposure to working fluids and to higher than ambient temperatures causes some shift in mechanical properties. However, to be considered for long-term service, the change in properties should be transient, and the material properties should reach equilibrium values. That is, material degradation should not continue as time goes on.

For this paper, our methods of evaluating material performance focus primarily on functional criteria as opposed to standard mechanical properties. For example, compressive stress relaxation is judged based on a seal's blowout pressure; ASTM designated tensile tests on dog bones have been omitted.

EXPERIMENTAL METHOD

Table 1 below summarizes the test conditions, and Fig. 1 shows a cutaway view of the test fixture. The two-piece fixture (Fig. 2) allows installation and removal of the top piston type seal without causing mechanical damage to the seal.

Table 1: Experimental Matrix

Variable	Description	
4 Coolant -Resistant	TFE/P, HNBR, EPDM, SILICONE	
Elastomers	(See appendix and Table 2 for details)	
1 Temperature	135°C	
4 Test Times	100, 250, 500, and 1000 Hours	
(In Addition to "Un-Aged" Samples)		
1 Test Fluid	Coolant, Fully Formulated (concentrate) Organic Acid Technology (OAT) Coolant- Heavy Duty	
Samples per Set of Test Conditions	1.274" ID x 0.139" Wall O-Rings (For piston seal);	
	Size 920 O-Rings (for bottom end cap seal)	
	Size 214 O-Rings (3 per fixture) Under 25% Compression (ASTM D1414)	
	Spherical Test Specimen	
	Torsion Rectangular Sample	

The test fixture of Fig. 1 was designed to closely simulate conditions of cooling system seals. Various components are submerged in coolant fluid during aging. The two-piece top plug is used for ease of installation and removal of the o-ring that seals the top of the cylinder in a piston seal configuration. During aging this o-ring is under a nominal 13% radial compression. A cutaway view of the fixture is shown in Fig. 2 below. There is no o-ring in the top cap of the fixture (i.e. the top of the piston seal is subject to dry heat and coolant aging). Also in the cylinder is a stack of three dash number 214 o-rings, each under 25% compression. The compressive stress relaxation (CSR) fixture (3) is also in the test cylinder. A cutaway view of the fixture and cross sectional view of the sample are shown in Fig. 3 and Fig. 4 respectively. Finally, the torsion rectangular sample sits in the test fluid as well. All rubber parts/samples in a given fixture were manufactured from the same rubber compound.

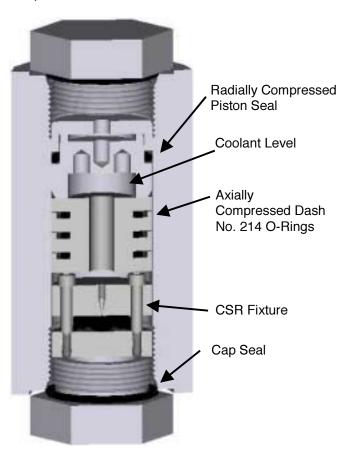


Figure 1: Test Cylinder Schematic

The assembled test cylinder is placed in an oven and aged for a specified amount of time at 135°C.

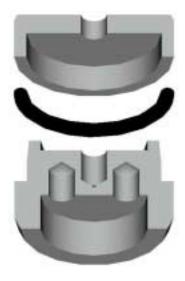


Figure 2: Piston Seal Schematic

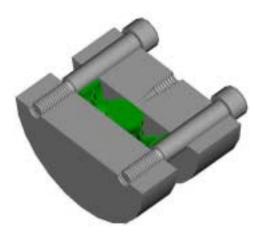


Figure 3: CSR Fixture Schematic

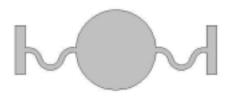


Figure 4: CSR Sample Cross Section

The base polymers of compounds tested are hereafter referred to via the abbreviations in Table 2.

Table 2: Material Designations

Trade Name	Polymer	D1418 Designation
AFLAS ®	TFE/P	FEPM
Therban®	HNBR	HNBR
Royalene®	EPDM	EPDM
SE4170X19D1	SILICONE	VSi

RESULTS

Aging of all samples took place at 135°C.

PISTON SEAL O-RINGS - RADIALLY COMPRESSED

Non-standard sized o-rings (author's part number 2785) were used as piston seals for the top of each fixture used (as shown in Fig. 2). Each data point shown represents a single seal (i.e. no replicates of this specimen were used). These seals were exposed on one side to dry heat aging, while exposed to coolant on the other. Data for compression set, tensile strength, ultimate elongation, and durometer of these samples are shown in figures 5 through 8 respectively.

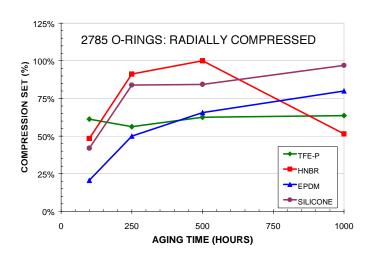


Figure 5: Radial Compression Set vs. Aging Time

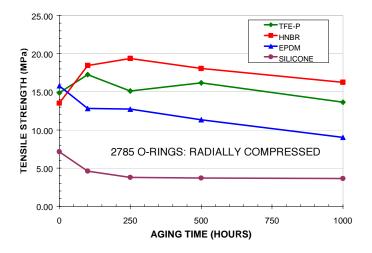


Figure 6: Tensile Strength vs. Aging Time

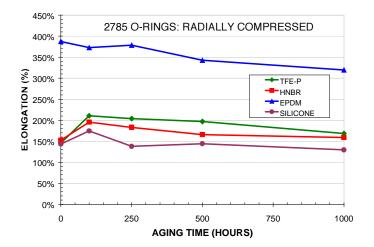


Figure 7: Ultimate Elongation vs. Aging Time

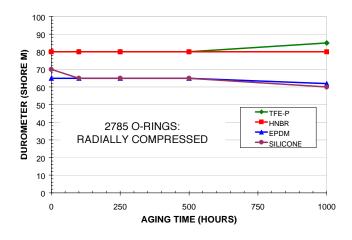


Figure 8: Shore M Durometer vs. Aging Time

AXIALLY COMPRESSED O-RINGS

Size –214 o-rings as shown in Fig. 1 were tested before and after aging. Test results include compression set, tensile strength, ultimate elongation, and durometer versus aging time; these are shown in figures 9 through 12 below.

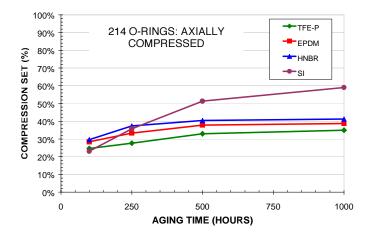


Figure 9: Compression Set vs. Aging Time

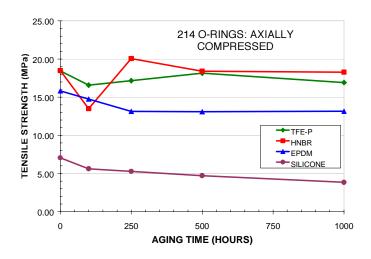


Figure 10: Tensile Strength vs. Aging Time

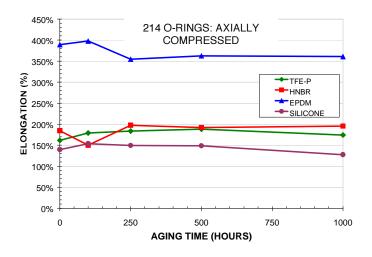


Figure 11: Ultimate Elongation vs. Aging Time

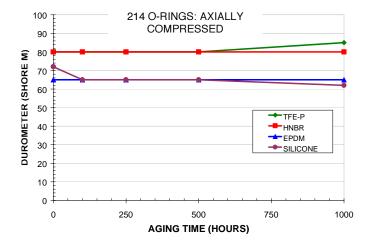
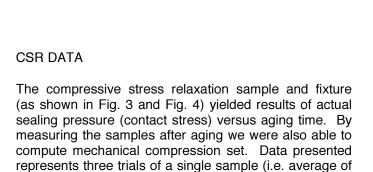


Figure 12: Shore M Durometer vs. Aging Time



three readings). These results are plotted in Fig. 13 and

Fig. 14.

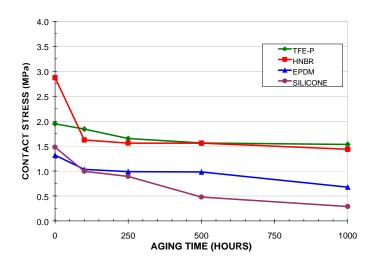


Figure 13: Retained Contact Stress vs. Aging Time

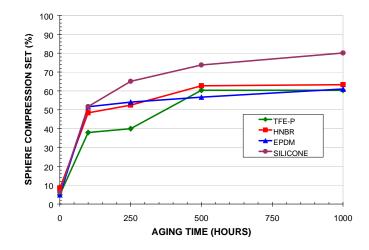


Figure 14: Sphere Compression Set vs. Aging Time

GLASS TRANSITION TEMPERATURE

Glass transition temperature, T_g , was calculated from plots of tan δ versus temperature. During the tests, temperature was increased at a rate of 5°C per minute; data was collected between -60°C and 80°C . Frequency of oscillation was 0.1 Hz. Amplitude of oscillation was chosen to be within the linear viscoelastic region (LVR) for each compound. The LVR was reevaluated after each aging time. Glass transition temperature versus aging time is plotted in Fig. 15.

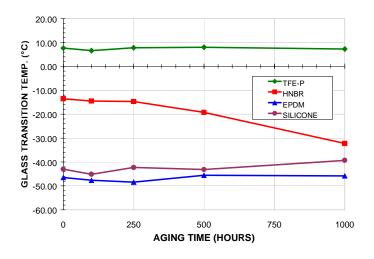


Figure 15: Glass Transition Temperature, $T_{\rm g}$ vs. Aging Time

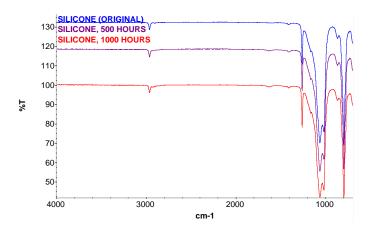


Figure 17: Interior Infrared Spectra for Silicone Aged 0, 500 and 1000 Hours

FTIR

Infrared spectra were collected for samples of the axially compressed o-rings tested. Scans of the surface of the seal (exposed to OAT fluid during aging) as well as the interior of the sample after slicing with a clean razor blade. Surface or EXTERIOR scans may include residue of the test fluid from direct contact; any shift in material INTERIOR spectra are due to chemical change of the elastomer and/or diffusion of the fluid through the surface into the bulk material.

Multiple spectra included on a single chart shown on an offset scale (i.e. they share the same relative scale for Transmittance, but not a common origin).

Data shown are for spectra collected using the Attenuated Total Reflectance (ATR) method- axes are percent transmittance versus wavenumber (cm⁻¹). Exterior and interior scans are shown for each material in figures 16 through 23 below.

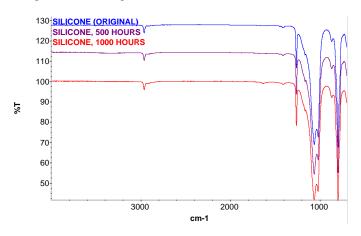


Figure 16: Exterior Infrared Spectra for Silicone Aged 0, 500 and 1000 Hours

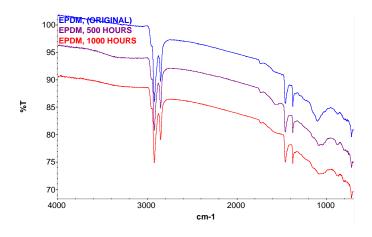


Figure 18: Exterior Infrared Spectra for EPDM Aged 0, 500 and 1000 Hours

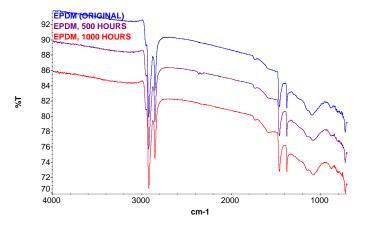


Figure 19: Interior Infrared Spectra for EPDM Aged 0, 500 and 1000 Hours

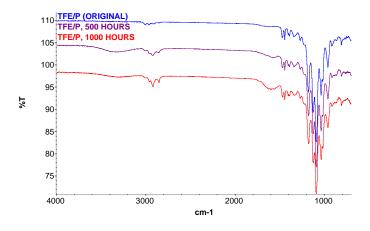


Figure 20: Exterior Infrared Spectra for TFE/P Aged 0, 500 and 1000 Hours

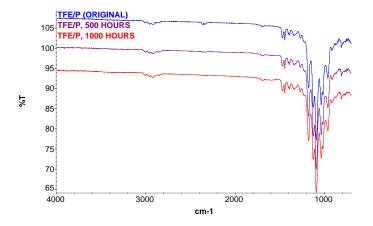


Figure 21: Interior Infrared Spectra for TFE/P Aged 0, 500 and 1000 Hours

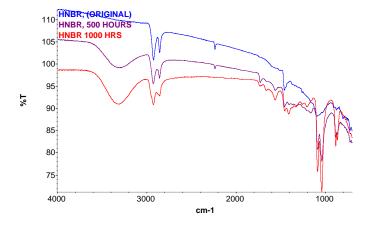


Figure 22: Exterior Infrared Spectra for HNBR Aged 0, 500 and 1000 Hours

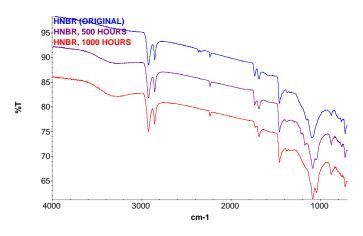


Figure 23: Interior Infrared Spectra for HNBR Aged 0, 500 and 1000 Hours

ANALYSIS

DYNAMIC MECHANICAL AND RHEOLOGICAL THERMAL ANALYSIS (DMRT) INTERPRETATION

Chemical attack by the fluids will logically take place at exposed surfaces of a seal and propagate through to the bulk of the material as diffusion and/or conversion of chemical species progresses. We thus assumed that the FT-IR surface changes would ultimately be representative of the elastomeric bulk phase. DMRT is a convenient and accurate way of measuring these changes. By reporting $G^{\prime\prime}$ (loss modulus) rather than G^{\prime} (elastic modulus), G^* (complex modulus) or tan δ , we have a better insight on network changes. Such network changes include an increase or decrease in crosslink density or chain scission.

FEPM and EPDM:

Temperature sweeps for material aged through 1000 hours show little variation from original; thus, plots are not included.

HNBR:

Temperature sweeps at each aging time (plotted in Fig. 24) show a progressive increase in $G^{\prime\prime}$ max through 500 hours. This is due to a diminishing very polar cyano group presence and its conversion to non-polar hydrocarbon groups. Material aged 1000 hours shows a dramatic shift in the $G^{\prime\prime}$ curve with glass transition temperature, T_g , approaching that characteristic of non-polar hydrocarbon elastomers.

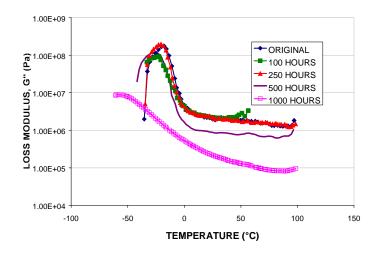


Figure 24: Loss Modulus vs. Temperature (HNBR)

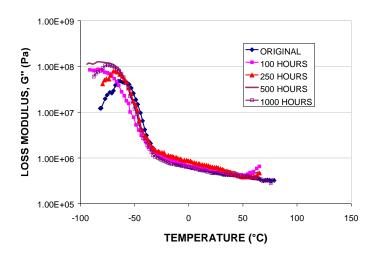


Figure 25: Loss Modulus vs. Temperature (SILICONE)

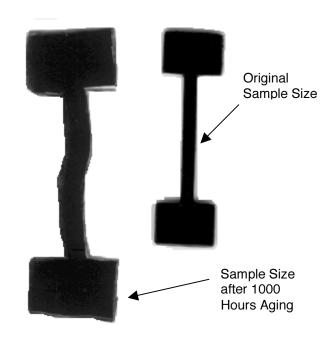


Figure 26: Swollen and Original DMRT Samples (HNBR)

SILICONE:

Temperature sweeps at each aging time (plotted in Fig. 25) show only minor changes from original; the maximum value of G" has increased slightly. This increase is consistent with the small amount of fluid uptake, which appears to be due to an alcohol presence.

FT-IR INTERPRETATION (4)

VSi (Silicone)

Silicone spectra are shown in figures 16 and 17.

There were no pronounced peaks with the exception of a relatively small peak at 2970 cm $^{-1}$, assignable to $-CH_2$ or $-CH_3$ bond stretching. The aged and unaged samples showed little to no difference in band widths/lengths. The peak at 1260 cm $^{-1}$ could be attributed to CH_3 -Si bond interactions. Peaks at 1062 cm $^{-1}$ and 1016 cm $^{-1}$ could be Si-O bond interactions as well. Overall, aged samples in OAT coolant differed only slightly from the unaged sample.

EPDM

Spectra for EPDM are shown in figures 23 and 24.

C-H bond stretching (symmetric & asymmetric) traditionally give rise to the peaks observed between 3000 cm⁻¹ to 2800 cm⁻¹, which would be expected with an EPDM structure. The next significant peaks are at 1460 cm¹ and 1375 cm⁻¹, which could be assigned to –CH₂ and –C-CH₃ interactions respectively. However, the spectrum did not change dramatically even after 1000 hours aging in OAT coolant.

TFE/P

Spectra for TFE/P are shown in figures 25 and 26.

C-H bond stretching (symmetric & asymmetric) gives rise to the observed peaks between 3000 cm⁻¹ and 2800 cm⁻¹. The intensity of these peaks changes significantly after 100 hours in OAT coolant. In the region between 3500 cm⁻¹ and 3000 cm⁻¹, there is an appreciable band development. That could be assigned to an OH- bond stretching due to alcohol. Absorption in the region of 1500 cm⁻¹ to 1400 cm⁻¹ is indicative of -CH₂ and -CH₃ groups. An absorption peak at 1080 cm⁻¹ could be assigned to a secondary aliphatic alcohol, C-O-Hstretching. From the ongoing, we can make an inference that TFE/P in OAT coolant at 100 hours produces/absorbs an OH- bond from the alcohol. At 250 and 500 hours, however, this band of OH- (alcohol) seems to be diminishing, and the hypothesis is that the alcohol is driven off as the aging time increases. If so, the overall effect of alcohol (be it positive or negative) is open for investigation.

In order to check the validity of our assumption of OH-alcohol, we first checked the OAT fluid (visually) to see if there were any impurities and or change in color. The aged and unaged fluids were then scanned to check if OH- alcohol absorption either diminished or increased, since that would be a good indicator of degradation.

We found the aged fluids to be unchanged, that is, the coloration of OAT coolant had not been altered significantly and no impurities were observed. The scans also showed no decrease in the OH-alcohol region throughout the aging history as well as the unaged fluid. From the ongoing, we can make another assumption that the OH- we observed on the TFE/P scans was a result of the OH-alcohol from OAT.

We ran another set of scans involving the interior and exterior of the TFE/P aged in OAT coolant. At 100 hours, it was clearly observed that the interior was significantly different from the exterior and the OH-alcohol observed on the TFE/P scan would have originated from the OH in OAT coolant (1,2-ethanediol). Therefore, the peak between 3000 - 3500cm⁻¹ was a result of residual OAT coolant on the surface of TFE/P and not a result of OH-alcohol diffusing/solvating into or reacting with the TFE/P.

<u>HNBR</u>

Spectra for HNBR are shown in figures 27 and 28.

There is characteristic -CH₂ and -CH₃ stretching between 3000 cm⁻¹ and 2800 cm⁻¹. Also present is the peak at 2230 cm⁻¹, and since this peak is present in all samples tested, we can assign that peak to -C≡N based on IR literature. At 100 hours, there is a band developing in the region of 3500 cm⁻¹ - 3000 cm⁻¹, indicating the presence of an alcohol. This bandwidth increases with aging time. There is also another peak developing at around 1596 cm⁻¹, which could be assigned a -COO-(ester/carboxylic acid), and this band increases with aging time. Another developing bandwidth is at 1723 cm⁻¹, and that is an indication of -COOR- interaction. Further down the spectrum, there are two developing peaks at 1089 cm⁻¹ and 1042 cm⁻¹ respectively. These peaks can be assigned to -C-O- interactions as a result of the alcohol being produced, that is, if we accept the bandwidth at 3300 cm⁻¹ to be an alcohol. These two peaks also increase with aging time and with increase in -OH- (alcohol) production.

After 1000 hours the HNBR surface showed a complete disappearance of the cyano group (−C≡N) with spectra indicating a strong presence of a secondary amide. The breakdown mechanism of the cyano group is in line with the predicted organic transformations detailed in Larock's Comprehensive Organic Transformations (5). The torsion rectangular specimen showed a dramatic (>50%) increase in volume after 1000 hours and there was a pronounced odor of ammonia in the OAT coolant.

ELASTOMER SUMMARY

FEPM

Sample/Test Type	Comments	
Piston seal o-ring,	Material is stable exposed to	
single sample, radial	OAT coolant /dry heat over 1000	
compression	hours (see figures 5-8).	
Size 214 O-Rings, 3	Material is stable over 1000	
samples, total	hours (see figures 9-12).	
immersion in OAT		
coolant, axial		
compression		
CSR Specimen	Contact Stress is stable over	
	1000 hours	
FTIR	Shows no significant changes	
	after 1000 hours at 135°C	
DMRT	Shows no significant changes	
	after 1000 hours at 135°C	

HNBR

Sample Type	Comments	
Piston seal o-ring, single sample, radial compression	Looking at results of most tests, material appears stable. However, the upward trend in compression set takes a rapid turn downward as a result of excessive swelling.	
	(See Fig. 26)	
Size 214 O-Rings, 3 samples, total immersion in OAT coolant, axial compression	Material appears stable, except in tensile strength.	
CSR Specimen	Contact stress is stable after initial decrease; swelling in OAT coolant masks chemical changes	
FTIR	Fig. 22 shows a disappearance of the cyano peak, indicating a chemical shift	
DMRT	Virtually total disappearance of G" response after 1000 hours shows substantial physical change	

EPDM

Sample Type	Comments
Piston seal o-ring, single sample, radial	Compression set and tensile strength are showing
compression	deterioration with aging time as a result of exposure to oxidative attack (see figures 5-7)
Size 214 O-Rings, 3 samples, total immersion in OAT coolant, axial compression	Material appears stable over 1000 hours
CSR Specimen	Material is showing a relatively strong downward trend in retained compressive stress after 1000 hours
FTIR	Shows no significant changes after 1000 hours at 135°C
DMRT	Shows no significant changes after 1000 hours at 135°C

SILICONE

Sample Type	Comments		
Piston seal o-ring,	Compression set still increasing		
single sample, radial	after 1000 hours; tensile		
compression	elongation and durometer		
	appear stable		
Size 214 O-Rings, 3	Tensile strength is deteriorating		
samples, total			
immersion in OAT			
coolant, axial			
compression			
CSR Specimen	Retained contact stress is still		
	decreasing after 1000 hours		
FTIR	Appears chemically stable		
DMRT	Shows a progressive increase in		
	G" low temperature value,		
	possibly due to alcohol uptake		
	from OAT coolant		

CONCLUSION

In summary, for long-term service in basic environments such as OAT coolant at 135°C, the HNBR and silicone (VSi) compounds tested have been shown to be incompatible. The silicone exhibits high stress relaxation over time. HNBR although appearing suitable by many tests, has serious chemical compatibility problems and high volume swell at longer times. The EPDM tested appears to be a viable option; however, as shown in previous work (1) (2), the material should not be exposed to dry heat, oil, or higher temperatures. The TFE/P elastomer tested has been shown to have the best performance characteristics in terms of long-term stability, chemical compatibility, and robustness (i.e. exposure to different environments, higher than anticipated temperature, dry heat, etc.) (1) (2).

ACKNOWLEDGMENTS

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CONTACT

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DEFINITIONS, ACRONYMS, ABBREVIATIONS

Compression Set:

Percent strain retained after aging for a given period of time. See ASTM D395 for a complete description.

DMRT:

Dynamic Mechanical and Rheological Testing/Thermal Analysis

FTIR:

Fourier Transform Infrared spectrometer or spectroscopy.

Strain Energy Density:

Amount of energy absorbed by an elastic body (per unit volume of material) in stretching. It is calculated by integrating the stress versus strain curve for a tensile specimen.

LVR:

Abbreviation for Linear Viscoelastic Region; range of strain amplitude for which a material's viscoelastic properties (e.g. storage and loss modulus) are independent of strain amplitude.

TFE/P:

Tetrafluoroethylene-propylene.

CSR

Compressive Stress Relaxation

OAT:

Organic Acid Technology Coolant; for this study, Texaco Extended Life Coolant (TELC)® was used.

APPENDIXCOMPOUND FORMULATIONS (PHR)

Ingredient/Polymer	Therban ®	Royalene ® 501	GE Silicone	AFLAS 100H
Compound	A3907		SE4170X19D1	7182X
Designation	HNBR	EPDM	SILICONE	Dipolymer
				(TFE/P)
AFLAS 100H				
THERBAN A3907	100.0			
ROYALENE ® 501		100		
GE Silicone				
Vulkanox ZMB-2	2.0		Proprietary	Proprietary
TE 80	1.0		Compound	Compound
FEF (N550) Black	25.0		of	of
MT (N990) Black	35.0		General Electric	Seals Eastern, Inc.
Paraplex G-50	10.0			
Varox DBPH-50	10.0			
Diak #7	4.0			
ZnO		5.0	_	
N774 Carbon Black		60.0		
Naugard® Q		1.0		
SR-350 Coagent		2.0		
DiCup® 40-KE		7.0		
Trilene® 77		10.0		

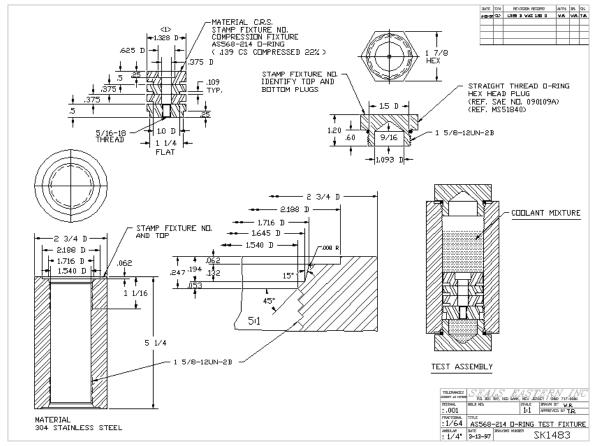


Figure 27: Test Vessel

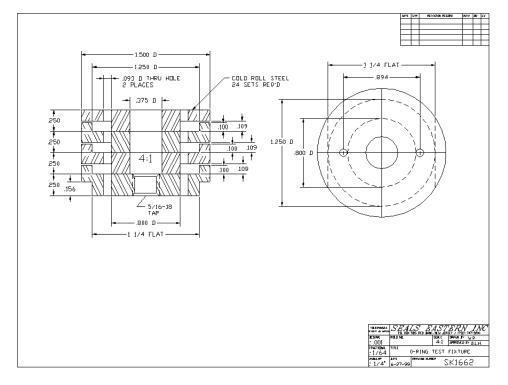


Figure 28: O-Ring Axial Compression Fixture

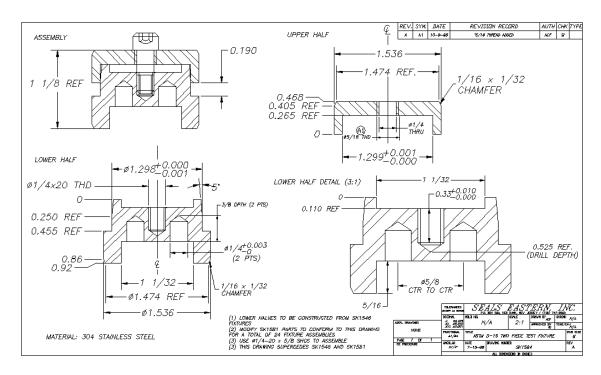


Figure 29: Two-Piece Fixture for Radial Compression of O-Rings

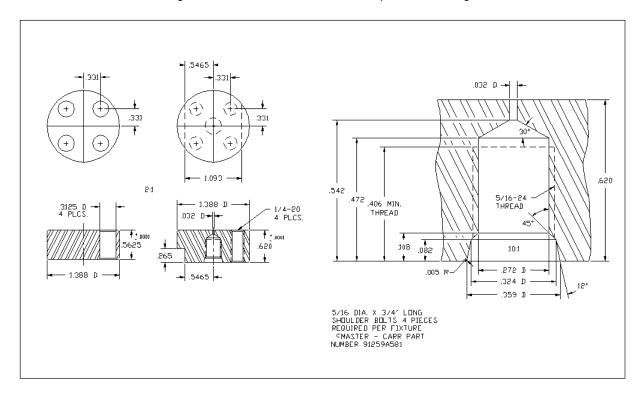


Figure 30: CSR Fixture