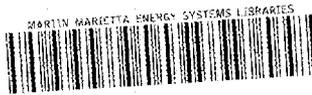


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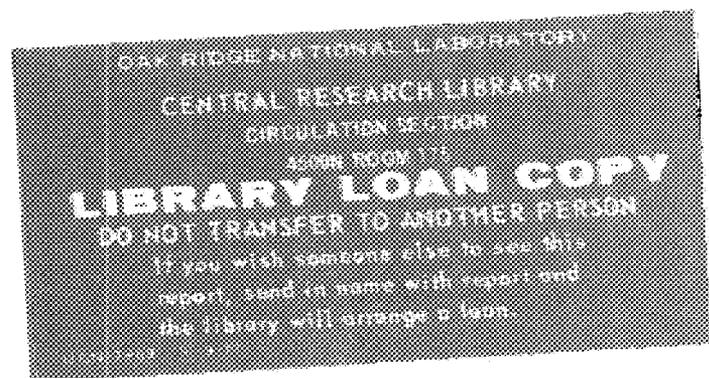


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ORNL/TM-10066

Evaluation of Selected Elastomer O-Ring Pump Seals for Service at the Wilsonville, Alabama, Advanced Coal Liquefaction Research and Development Facility

C. C. Skena
J. R. Keiser



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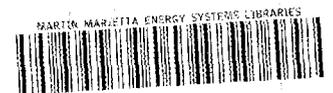
EVALUATION OF SELECTED ELASTOMER O-RING PUMP SEALS FOR SERVICE AT
THE WILSONVILLE, ALABAMA, ADVANCED COAL LIQUEFACTION
RESEARCH AND DEVELOPMENT FACILITY

C. C. Skena and J. R. Keiser

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C. C. Skena[†] and J. R. Keiser

ABSTRACT

Previous laboratory tests of elastomer O-rings in coal liquefaction solvents conducted at L'Garde, Inc., indicated that certain ethylenepropylenediene monomer (EPDM) compounds provided the best performance when a backup ring was used to limit swelling. Before service testing in a pump at the Wilsonville, Alabama, Advanced Coal Liquefaction Research and Development Facility, tests of six selected elastomers in the appropriate Wilsonville-produced solvent were conducted at Oak Ridge National Laboratory (ORNL). The ORNL tests measured the elastomers' changes in cross section, weight, density, and relative flexibility. Although two perfluoroelastomers showed less degradation of most properties during these tests, it was decided to proceed with service testing of two EPDM elastomers because of their much lower cost.

INTRODUCTION

Exxon Research and Engineering Company, Florham Park, New Jersey, has been searching for a commercially available elastomer with improved resistance to coal liquefaction pilot plant solvents. As part of that search, it sponsored a test program at L'Garde, Inc., Newport Beach, California, in which O-rings were tested by immersion and static simulation at elevated temperatures. L'Garde's report¹ concluded that Precision's[‡] ethylenepropylenediene monomer (EPDM) 42679 was superior to

*Research sponsored by the U.S. Department of Energy, Pittsburgh Energy Technology Center, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

[†]Now located at the Y-12 Plant, Development Division, Oak Ridge, Tenn.

[‡]Precision Rubber Products Corporation, Lebanon, Tenn.

DuPont's* costly Kalrez 1050 perfluoroelastomer in the simulation test. However, in the immersion test, the opposite conclusions were reached. On the basis of the superior performance of Precision's EPDM 42679 in the simulation test, Exxon engineers requested that the U.S. Department of Energy (DOE) conduct a field evaluation program in pumps at the Wilsonville, Alabama, Advanced Coal Liquefaction Research and Development Facility.² Because Oak Ridge National Laboratory (ORNL) has been providing technical support to the Wilsonville facility since 1979, we were subsequently requested to conduct hot immersion tests on the elastomer O-rings before service testing in Wilsonville's flush solvent pump P1039. This pump circulates hydrotreated process solvent and operates at 177 to 232°C (350-450°F).³ If the elastomer O-rings are found to be acceptable for service in the P1039 pump, they will be considered for evaluation in a more difficult application, the P119 vacuum tower bottoms pump, which operates at 316 to 338°C (600-640°F). The objective of the present study was to determine, by elevated-temperature immersion tests, which O-rings (if any) show promise for use in the P1039 pump.

MATERIALS

Information on the O-ring compounds selected for this study is presented in Table 1. The O-ring compound selection was influenced by the earlier work sponsored by Exxon.¹ In addition to the three EPDM compounds (Parker† E692-75 and E962-85 and Precision 42679), one fluoroelastomer (Precision Viton 16609) and two perfluoroelastomers (DuPont Kalrez 1050 and 4079) were included in the study (Table 1). To establish a frame of reference, the currently used shaft seal in the P1039 pump (Durafite marketed by Durametallic‡) was included in the study. The Durafite seal is not as flexible as the other seals tested, and it is made from layers

*DuPont Company, Elastomer Chemicals Department, Wilmington, Del.

†Parker Seals, O-Ring Division, Lexington, Ky.

‡Durametallic Corporation, Kalamazoo, Mich.

Table 1. Information on O-ring seals

Manufacturer ^a	Identification or compound number	Trade name	Generic name
Control seal^b			
Durametallic	PDGD-1750333	Durafite	Graphite
Test O-rings			
Parker	E692-75		EPDM ^c
Parker	E962-85		EPDM
Precision	42679	Nordel 1660	EPDM
Precision	16609	Viton	Fluoroelastomer
DuPont	1050	Kalrez	Perfluoroelastomer
DuPont	4079	Kalrez	Perfluoroelastomer

^aExcept for the Kalrez compounds, all the O-rings tested were nominally 4.45-cm (1.75-in.) ID and 5.40-cm (2.125-in.) OD, which corresponds to a No. 327 size O-ring.

^bCurrently used shaft seal in P1039 pump.

^cEthylenepropylenediene monomer.

of graphite bonded together in a rectangular cross section. The EPDM O-rings are much lower in cost than the currently used Durafite seal or the Kalrez seals.

The Wilsonville facility provided about one gallon of the V1066 hydrotreated process solvent from each of two Wilsonville runs for the ORNL tests. The nitrogen and sulfur contents, boiling point ranges, and specific gravities of the solvents are shown in Table 2. The solvent from the most recent Wilsonville run (No. 247) had a higher nitrogen content, boiling temperature range, and specific gravity than the solvent from the No. 246 run (Table 2). In addition, the average boiling point of the No. 247 solvent was 41°C higher than that of the No. 246 solvent. The variations in boiling points for the various polynuclear aromatic hydrocarbons and other components found in the two samples of V1066 solvent are best shown in Fig. 1. The differences in the solvents might be expected to cause some variation in the test results, but we believe the solvents were sufficiently similar that solvent-related variations in test results

Table 2. Selected properties of V1066 hydrotreated process solvent^a

Sample identification	Element		Boiling point (°C)			Specific gravity	Total aromatics (%)
	N (%)	S (ppm)	Initial	Final	Average		
Run No. 246 at 10:35 on 5/8/84 (SN No. 29367). Used to test all but the Kalrez O-rings.	0.12	507	184	399	317	0.9483	32
Run No. 247 at 9:45 on 1/4/85 (SN No. 43348). Used to test only the Kalrez O-rings.	0.18	422	232	406	358	0.9929	37

^aThe solvents were obtained from the Wilsonville, Alabama, Advanced Coal Liquefaction Research and Development Facility.

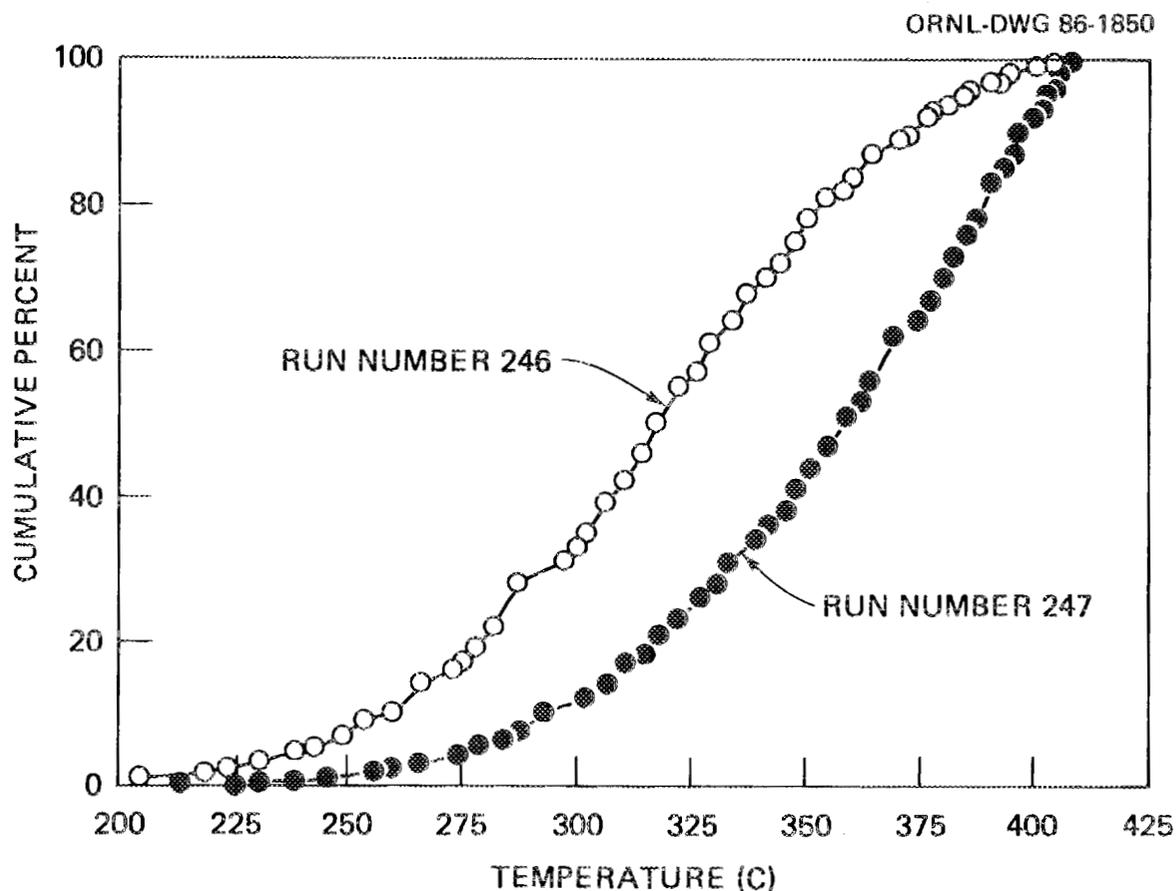


Fig. 1. Variations in boiling points of the two lots of V1066 hydrotreated process solvent used in the hot immersion tests.

are insignificant. Because the Kalrez O-rings were obtained after the start of the project when the supply of No. 246 oil was depleted, only those O-rings were tested in the No. 247 solvent; therefore, solvent-caused variations in the results would only be a consideration when comparing Kalrez with any of the other compounds.

EXPERIMENTAL WORK

Two O-rings of each compound were immersion tested in the V1066 hydrotreated process solvent for 72 h at 204°C (400°F). The tests were conducted in a manner similar to that outlined in ASTM Designation: D471-79.⁴ A separate test run was made for each O-ring compound, and 600 cm³ of virgin solvent was used for each run to avoid any possibility of contamination from other O-rings or depletion of solvent components, which would influence the test results. Seven runs were made in the ORNL-designed and -built glass test apparatus shown in Fig. 2. The large opening at the top of the test vessel enabled removal of the swollen O-rings without deflection or stressing. The very large Kalrez 4079 O-rings [11.43-cm (4.5-in.) OD] were spirally wound to fit the test apparatus. A water-cooled condenser was attached to the test vessel (at the ground glass joint) to minimize loss of volatile materials in the process solvent. Six thermocouples were used to control, monitor, and record the temperature at various locations in the system.

The volume, dimensions, weight, density, and relative flexibility of the O-rings were determined before and after testing. The volume of each O-ring was determined by water displacement. Dimensions were established by micrometer measurements. Weights were measured on a Mettler AK160 balance. Densities were calculated from the measured weights and volumes.

To determine the relative flexibility of the O-rings, a 200-g weight (200.5 g including a wire hook) was suspended from each O-ring, and the major axis (outside of the ellipse that formed) was measured and recorded. The outside toroidal diameter of each O-ring in the unstressed horizontal position was subtracted from that of the 200-g-stressed outside major axis to yield the deflection before and after testing. Deflections of the

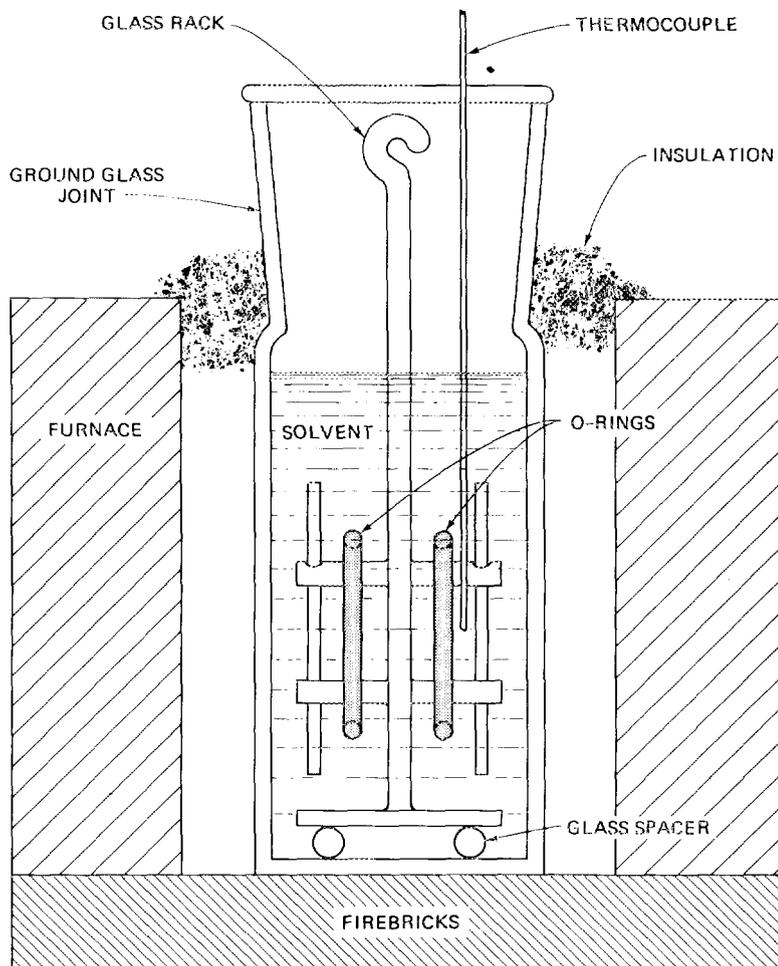


Fig. 2. Apparatus for hot immersion testing of O-rings.

O-rings were compared to establish relative flexibility. The relative flexibility of the Kalrez O-rings could not be determined with this test method because the manufacturer cut through their cross sections before providing them for the study.

The testing procedure consisted of placing the O-rings in the solvent at ambient temperature, heating to 204°C (400°F) (about 2 h was required), holding at 204°C for 72 h, allowing the vessel to cool to less than 66°C (150°F) (about 2 1/2 h was required with the top insulation removed), and removing the O-rings from the test vessel and placing them on paper towels for a minimum of 12 h to absorb the excess solvent before weighing and measuring them.

RESULTS AND DISCUSSION

SWELLING OF O-RINGS

Photographs of the O-rings before and after hot immersion testing are shown in Figs. 3 through 9, and the volume and dimensional changes are indicated in Tables 3 and 4, respectively. It is obvious that major differences exist in the amount of swelling (Fig. 10). Kalrez 1050 had the lowest volume change, followed by (in order of increasing volume change) Kalrez 4079, Viton, Durafite, Precision EPDM 42679, and Parker EPDMs E962-85 and E692-75. The Parker EPDM E692-75 O-ring swelled so much that an untested O-ring could be passed through a tested O-ring, an increase in the average inside toroidal diameter of more than 30%, whereas the inside toroidal diameter of the Durafite seal increased by only 0.1% (Table 4). As shown in Table 3, the volume of the Durafite seal increased by about 13.8%, primarily from swelling in the thickness (axial) direction (Table 4). Because swelling in the thickness direction would be taken up by the compression springs in the Durametalllic Corporation seal, it may not be a disadvantage. In addition, swelling in the thickness dimension may be less in actual service because of the compressive spring loading. The average radial cross section of the Durafite seal increased by only 1.3%, whereas the thickness of the rectangular cross section of the Durafite seal increased by 10%, clearly showing that the swelling tendency is anisotropic. The average inside and outside diameters of the currently used Durafite seal showed the least swelling (<0.25%) of all the seals for which reliable diametral measurements could be made (all but Kalrez) (Table 4).

In general, the swelling of the elastomer O-rings was reasonably isotropic (Table 4). The relative swelling characteristics of the O-rings are best shown in Fig. 10. All the EPDM O-rings swelled significantly, ranging from 81 vol % for the Precision 42679 to about 123 vol % for the Parker E692-75. The average increases in radial cross-section diameter for these two EPDM O-rings was 1.17 mm (0.046 in.) and 1.62 mm (0.064 in.), respectively. The other EPDM O-ring in the study, Parker E962-85, swelled by about 94 vol %, and its cross section increased by 1.37 mm (0.054 in.) (Fig. 10).

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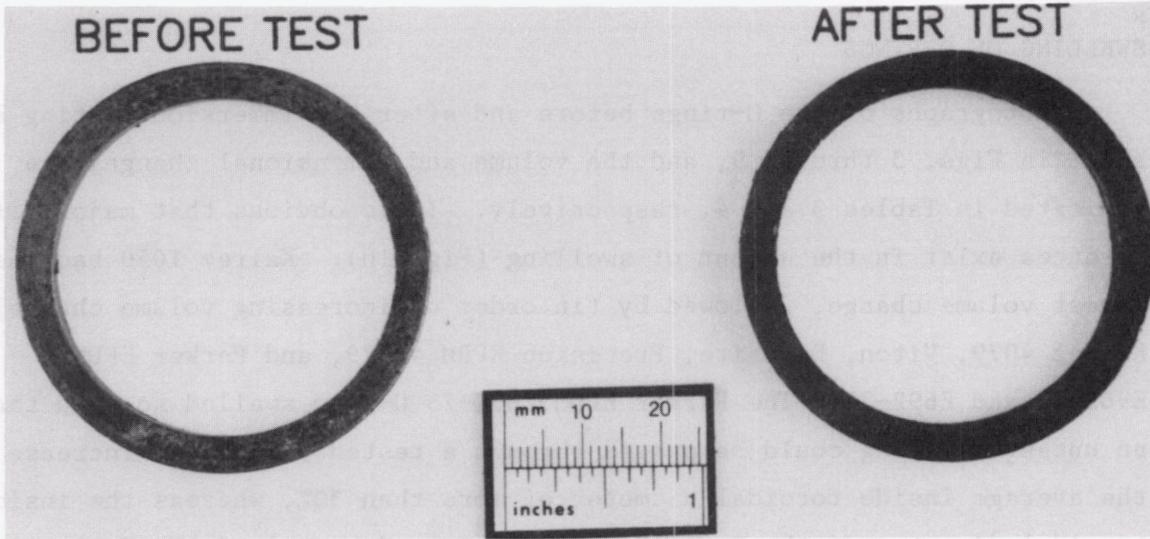


Fig. 3. Appearance of Durametalllic Durafite O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated in process solvent.

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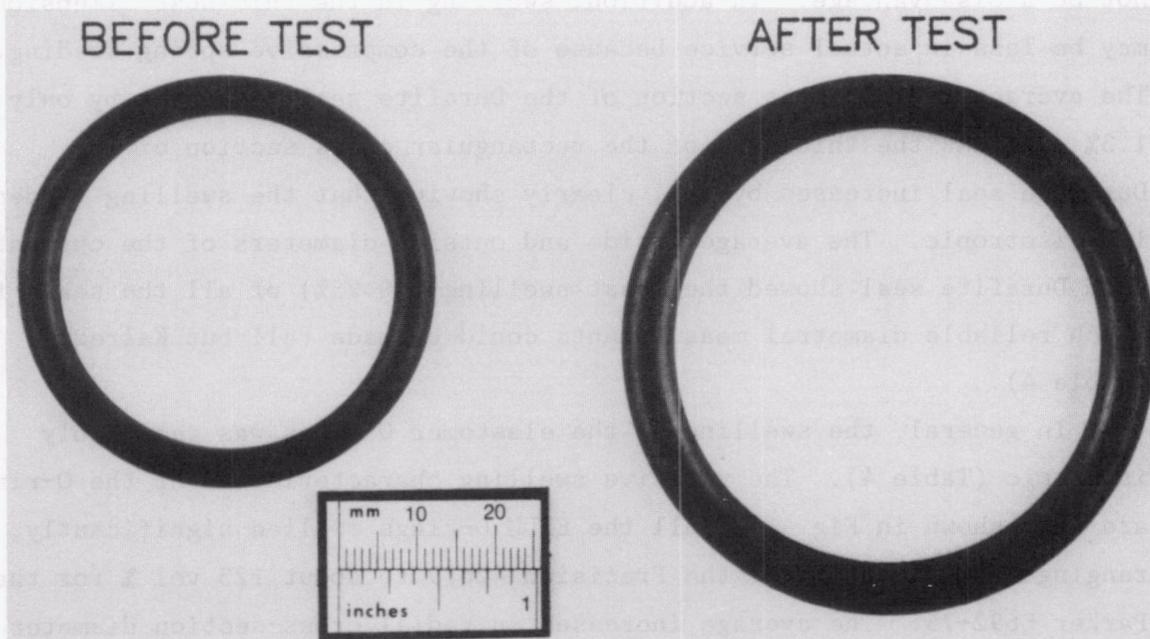


Fig. 4. Appearance of Parker E692-75 EPDM O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent.

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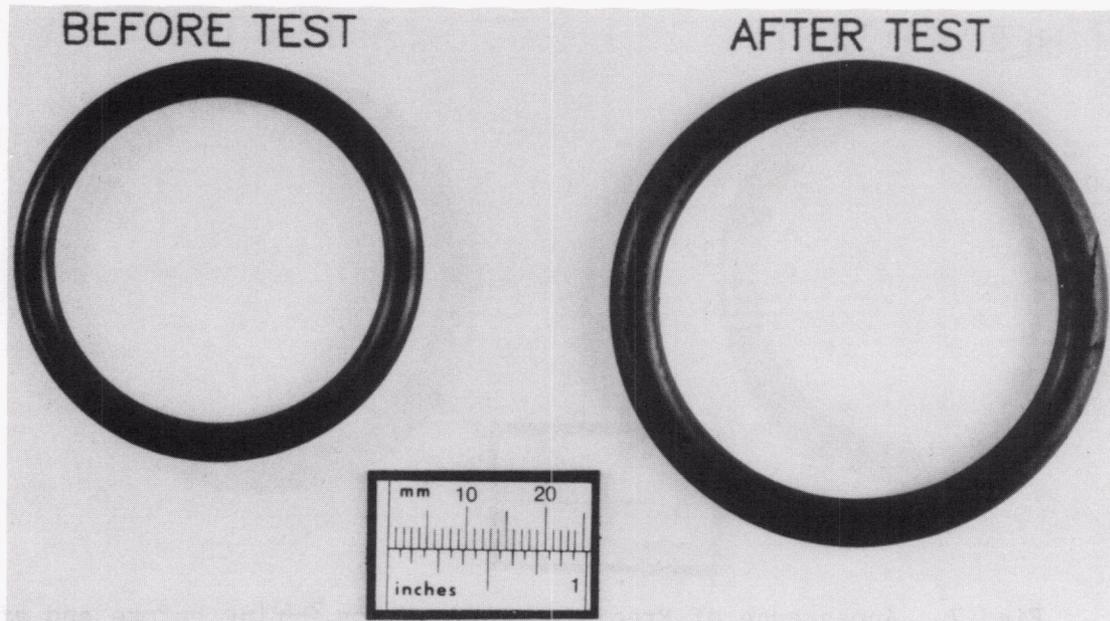


Fig. 5. Appearance of Parker E962-85 EPDM O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent.

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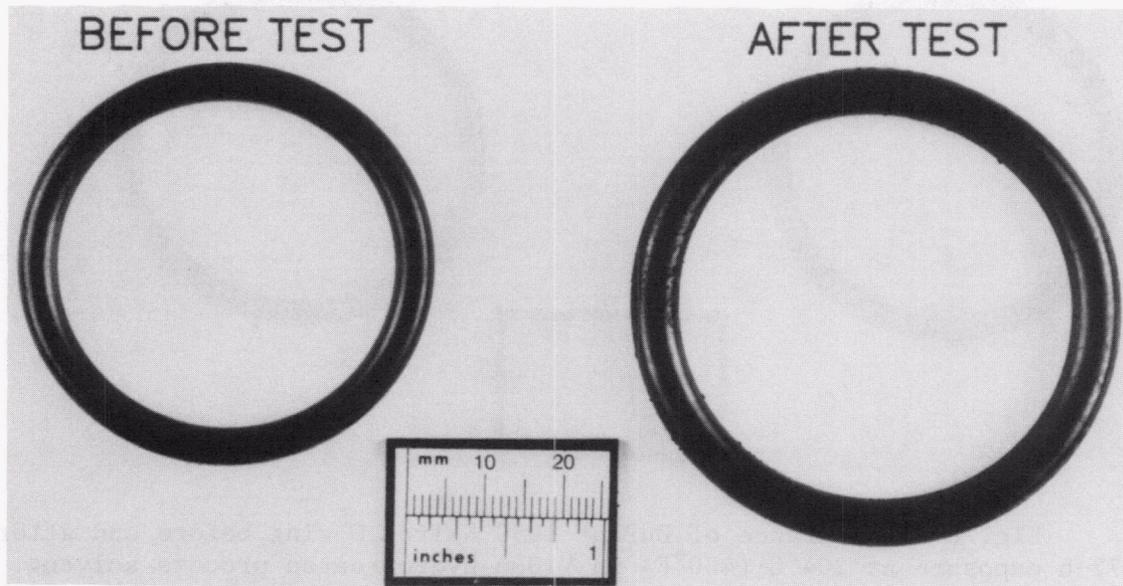


Fig. 6. Appearance of Precision 42679 EPDM O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent.

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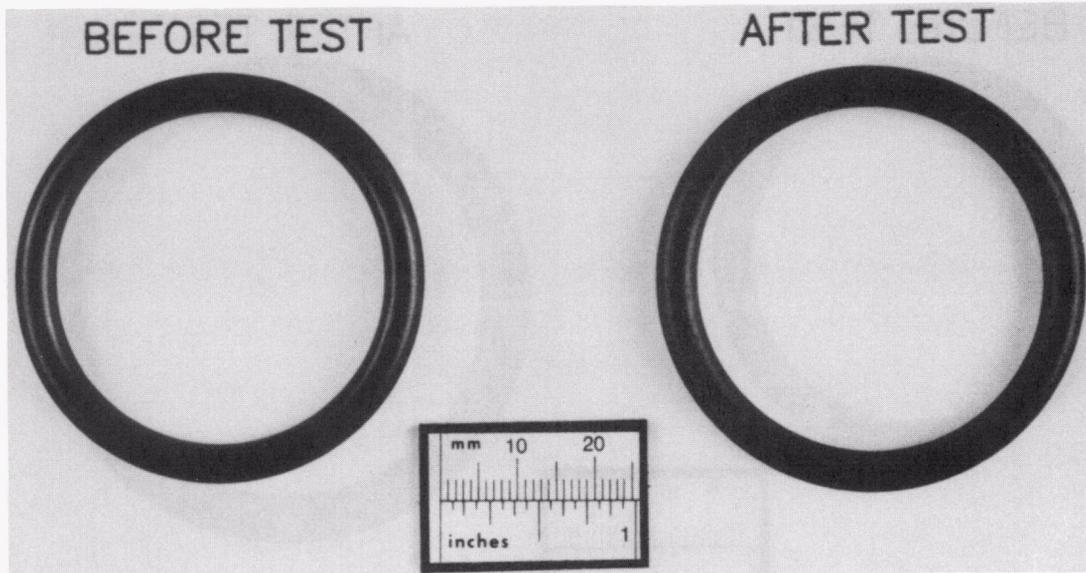


Fig. 7. Appearance of Precision 16609 Viton O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent.

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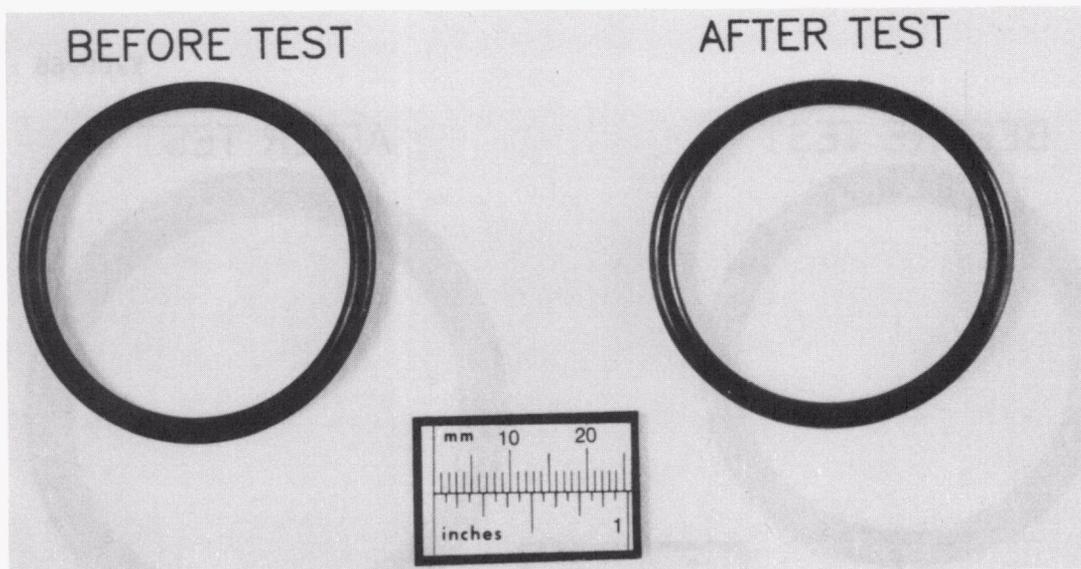


Fig. 8. Appearance of DuPont 1050 Kalrez O-ring before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent.

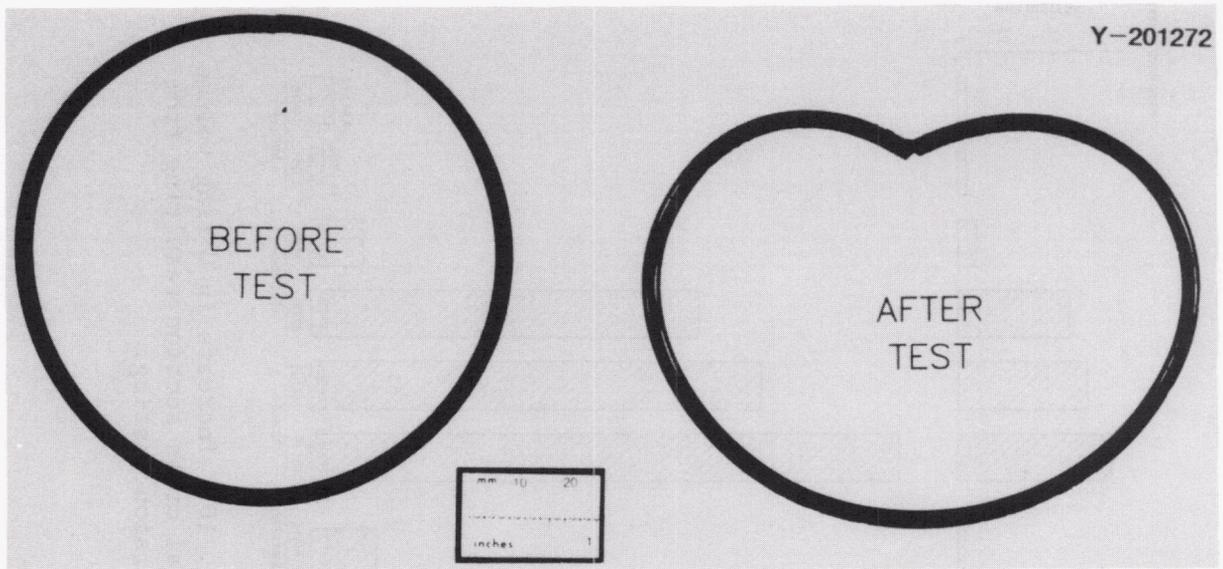


Fig. 9. Appearance of DuPont 4079 Kalrez O-rings before and after 72-h exposure at 204°C (400°F) in V1066 hydrotreated process solvent. These O-rings were much larger in diameter than the other O-rings tested and were cut by the manufacturer because they had fabrication defects. Testing in our assembly required winding them into a small coil, thus causing the deformation apparent in the O-ring on the right in the photograph.

Table 3. Volume increase of O-rings resulting from hot immersion testing

Identification ^a	Volume change ^b (cm ³)	Volume increase (%)	Identification ^a	Volume change ^b (cm ³)	Volume increase (%)
Durametallic			Precision		
Durafite	0.42	13.8	Viton 16609	0.37	10.6
Parker			DuPont		
EPDM E692-75	4.27	122.6	Kalrez 1050	0.10	2.6
Parker				0.03	
EPDM E962-85	3.10	93.7	DuPont		
Precision			Kalrez 4079	0.33	4.9
EPDM 42679	2.68	80.7		0.16	

^aExcept for the Kalrez O-rings, all the O-rings tested were nominally 4.45-cm (1.75-in.) ID and 5.40-cm (2.125-in.) OD, which corresponds to a No. 327 size O-ring.

^bValues given are the average for two O-rings except for Durafite (one O-ring was tested) and Kalrez (two O-ring sizes were used for each test, and the volume change could not be averaged).

Table 4. Dimensional change of O-rings resulting from hot immersion testing

Identification ^a	Cross section change (%)	Inside diameter change (%)	Outside diameter change (%)
Durametallic Durafite	1.3 (radial) 10.0 (axial)	0.1	0.2
Parker EPDM E692-75	30.2	31.4	29.6
Parker EPDM E962-85	25.8	22.6	23.4
Precision EPDM 42679	22.2	19.2	20.1
Precision Viton 16609	3.3	3.3	4.4
DuPont Kalrez 1050	1.4	<i>b</i>	<i>b</i>
DuPont Kalrez 4079	1.8	<i>b</i>	<i>b</i>

^aExcept for the Kalrez O-rings, all the O-rings tested were nominally 4.45-cm (1.75-in.) ID and 5.40-cm (2.125-in.) OD which corresponds to a No. 327 size O-ring.

^bManufacturer had cut O-rings, so overall ID and OD could not be measured accurately after exposure.

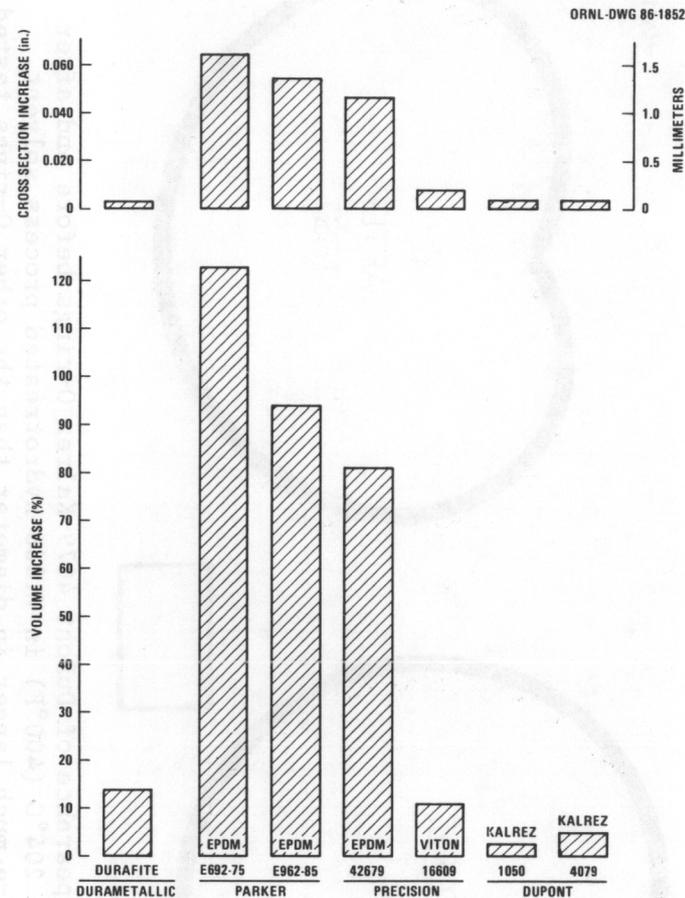


Fig. 10. Increase in O-ring volume and radial cross section resulting from hot immersion testing.

Clearly, the DuPont Kalrez O-rings had the smallest volume increases, averaging about 3 and 5% for Kalrez 1050 and 4079, respectively (Fig. 10). The radial cross sections of both Kalrez O-rings increased by about 0.076 mm (0.003 in.) or 1.44%, which is only slightly greater than the increase of the Durafite seals.

WEIGHT AND DENSITY CHANGES

Although the weight and density of the seals are not of any significance in themselves, changes in those parameters may indicate the elastomer's ability to resist attack by the solvent. The weight and density changes resulting from hot immersion testing are shown in Table 5 and Fig. 11. The weight of all the O-rings increased during the tests, ranging from about 1.26% for the Kalrez 1050 to about 93% for the Parker EPDM E692-75 (Table 5). Of the EPDM O-rings, the Precision 42679 showed the smallest weight gain, about 67%. The weight of the Durafite seal increased by about 60%.

The density of the Durafite seal increased by about 39% during the test, whereas the density of the elastomer O-rings decreased, ranging from 1.94% for the Kalrez 1050 to about 13% for the Parker E692-75 EPDM. Of the EPDM O-rings investigated, the Precision 42679 was least changed, about 8%.

RELATIVE FLEXIBILITY

Deflections of the O-rings while supporting a 200-g weight (Table 6) are plotted in Fig. 12 to indicate the relative flexibility before and after the tests. The Durafite seal is not shown in Fig. 12 because it did not deflect before or after testing. Of the EPDM O-rings, the Precision 42679 showed the least deflection before and after testing on an absolute basis (Fig. 12). However, on a percentage change basis, deflection of the Precision 42679 EPDM was greatest at 717% (Table 6). On an absolute basis, the deflection of the Parker E692-75 was affected most by the hot immersion test. The Viton O-ring showed a significant reduction in deflection after testing. Although the deflection of the Kalrez O-rings could not be measured, hand flexing them after testing and comparing them

Table 5. Weight and density changes in O-rings resulting from hot immersion testing

Identification ^a	Weight ^b		Density	
	Before test (g)	Increase (%)	Before test (g/cm ³)	Change (%)
Durametallic				
Durafite	3.1038	59.8	1.05	+39.0
Parker				
EPDM E692-75	4.3730	93.0	1.26	-13.3
Parker				
EPDM E692-85	3.7882	78.1	1.14	-8.1
Precision				
EPDM 42679	3.8045	66.7	1.15	-7.8
Precision				
Viton 16609	7.0676	4.0	2.02	-6.0
DuPont				
Kalrez 1050	7.2774	1.3	2.04	-1.9
Kalrez 4079	2.7653			
Kalrez 4079	15.2957			
Kalrez 4079	6.0513	1.9	2.03	-2.8

^aExcept for the Kalrez O-rings, all the O-rings tested were nominally 4.45-cm (1.75-in.) ID and 5.40-cm (2.125-in.) OD, which corresponds to a number 327 size O-ring.

^bValues given are the average for two O-rings except for Durafite (one O-ring was tested) and Kalrez (two O-ring sizes were used for each test, and the volume change could not be averaged).

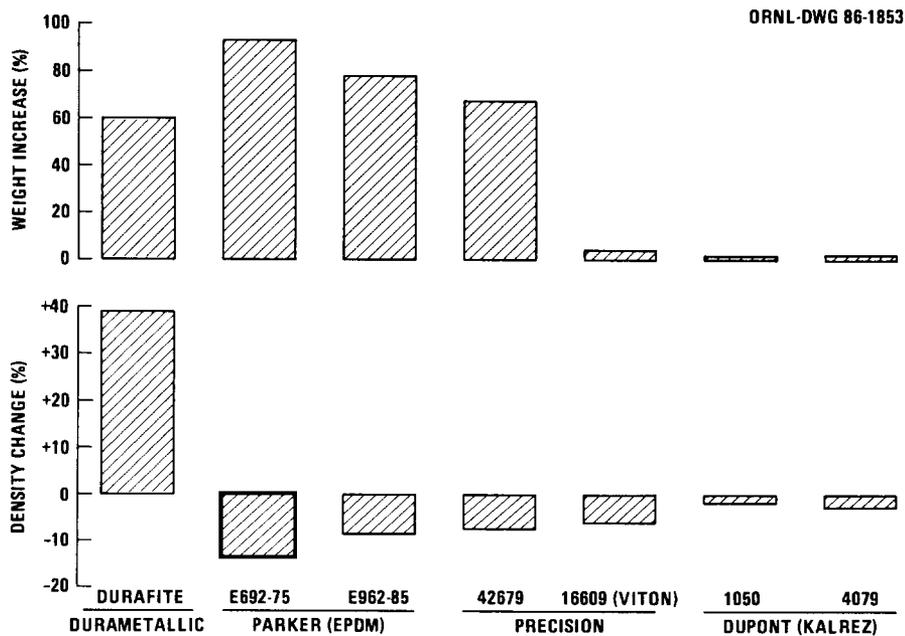


Fig. 11. Changes in O-ring weight and density resulting from hot immersion testing at 204°C in V1066 process solvents.

Table 6. Deflection change of O-rings after hot immersion testing

Identification ^a	Deflection ^b		Change (%)	Identification ^a	Deflection ^b		Change (%)
	Before test (mm)	After test (mm)			Before test (mm)	After test (mm)	
Durametallic Durafite	0.000	0.00	0	Precision Viton 16609	2.6	0.2	-92
Parker EPDM E692-75	6.8	21.5	+218	DuPont Kalrez 1050	All the Kalrez O-rings were judged to be slightly <u>less</u> flexible after testing.		
Parker EPDM E962-85	1.9	9.9	+425	DuPont Kalrez 4079			
Precision EPDM 42679	1.0	8.5	+717				

^aExcept for the Kalrez O-rings, all the O-rings tested were nominally 4.45-cm (1.75-in.) ID and 5.40-cm (2.125-in.) OD, which corresponds to a No. 327 size O-ring.

^bSupporting a 200.5-g weight.

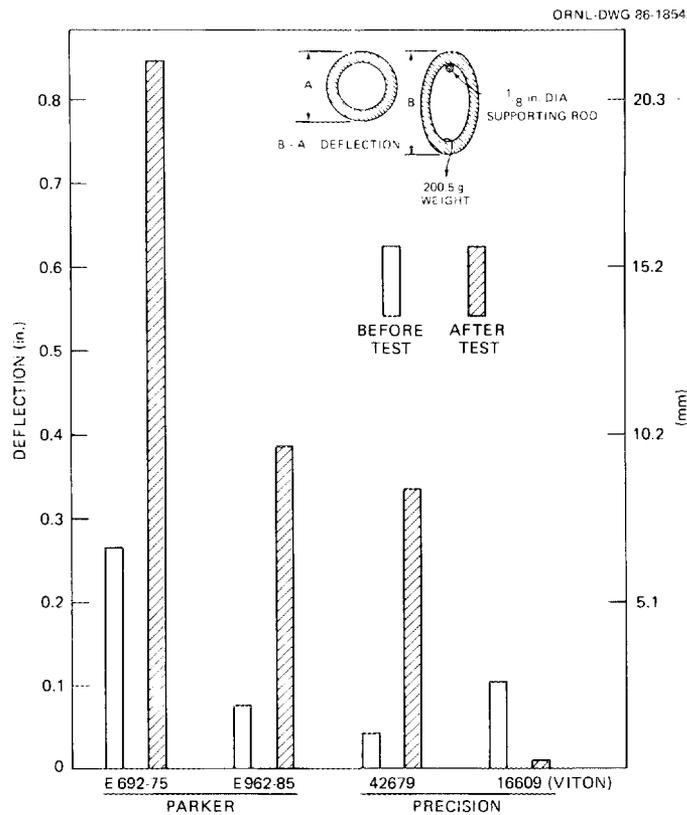


Fig. 12. Relative flexibility of O-rings before and after tests.

with as-received O-rings of the same size indicated that their flexibility was slightly reduced by the test. About one month after testing, one O-ring from each set was held by hand and twisted to gain additional knowledge of the flexibility and nature of the materials. All of the EPDM O-rings were very flexible, whereas the Viton O-ring was very stiff, readily cracked in numerous places where tensile stress was applied, and easily broken. The Durafite was rigid, but it readily delaminated in several places before breaking. Visual and low-power microscopic examinations suggest that the Durafite seal is laminated of numerous layers bonded to form a single unit.

POROSITY

While determining the volume of the O-rings by water immersion before testing, it was found that only the Durafite seal absorbed water. After testing, all the O-rings absorbed water. It appears that the swelling of the elastomer O-rings is, at least in part, related to porosity caused by the hot immersion tests. As shown in Fig. 13, the volume increase is directly proportional to the weight gain for the elastomer O-rings. Apparently, the weight gain results from the solvent permeating and filling the pores created by the elevated-temperature tests. Therefore, the density of the elastomer O-rings decreases (Fig. 11) because the specific gravity of the solvent (ranging from 0.9483 to 0.9929 g/cm³) is lower than the specific gravity of the O-rings (ranging from 1.05 to 2.04 g/cm³). Note that the Durafite seal did not swell in proportion to its weight gain, indicating that the porosity existed before testing. This finding is supported by the observation that the Durafite seal absorbed water before testing.

WILSONVILLE DATA

Wilsonville plant personnel also conducted elevated-temperature immersion tests on the subject O-rings,⁵ and the resulting data are plotted in Fig. 14. Although immersion times and temperatures used in the Wilsonville and ORNL studies differed, the test results for swelling and weight gain were similar. The Wilsonville data clearly show that, in

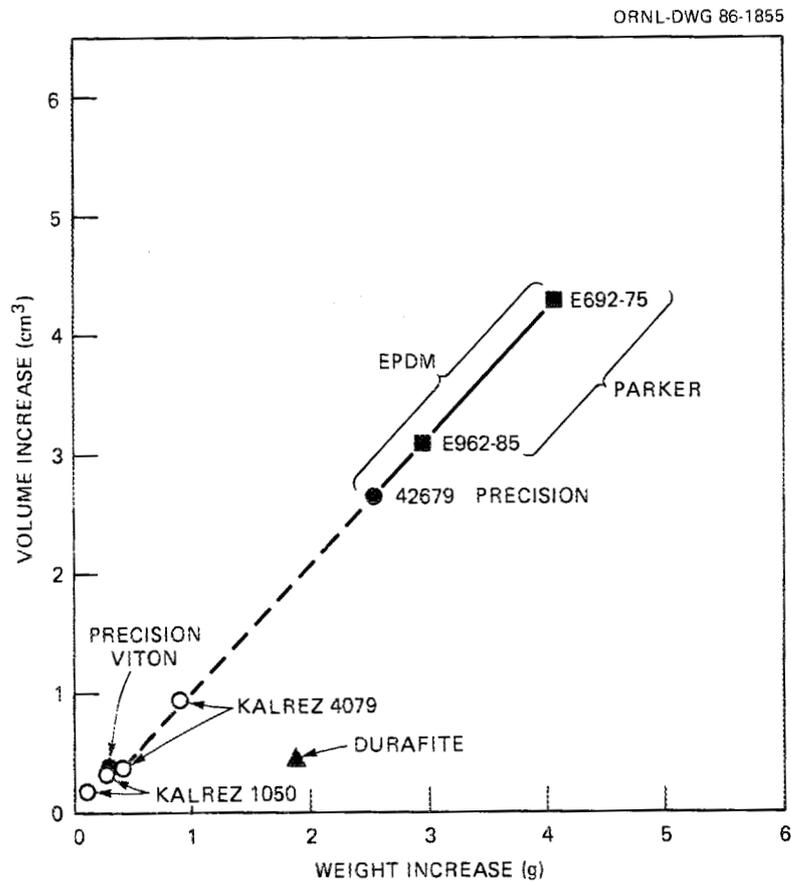


Fig. 13. Relationship between volume and weight increase after hot immersion testing.

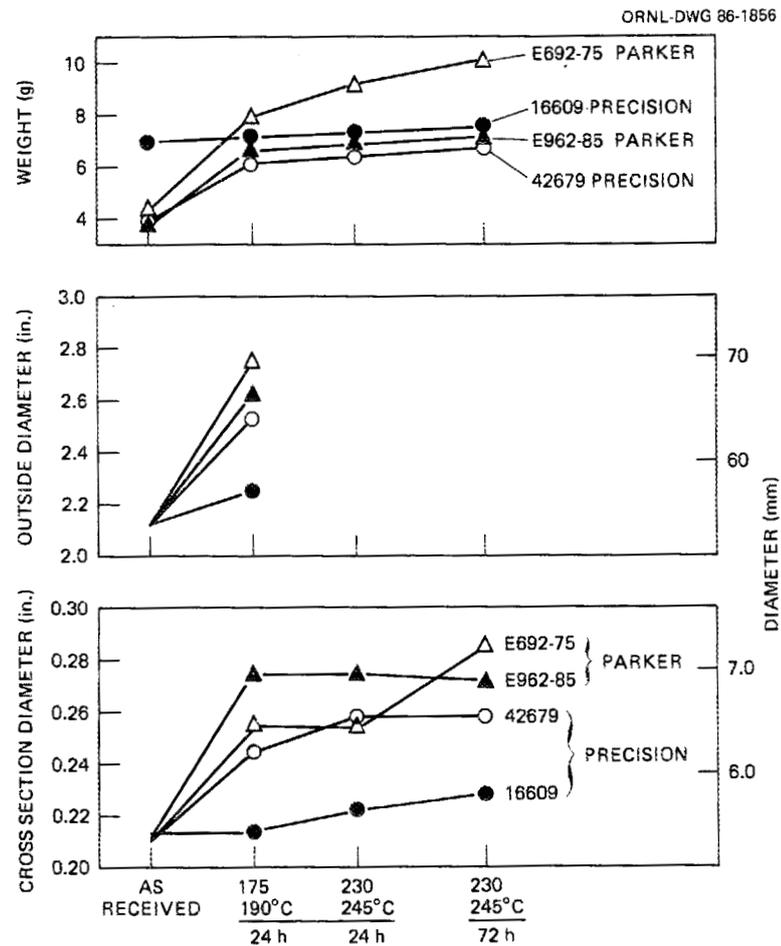


Fig. 14. Effect of temperature and time in hydrotreated process solvent (from P1039 pump) on elastomer O-rings from Precision and Parker in tests conducted by Catalytic, Inc., engineers at Wilsonville, Alabama.

general, swelling and weight gain of the EPDM O-rings occurred most rapidly during the first day of testing despite the relatively low temperature, 177 to 191°C (350-375°F).

SUMMARY

Changes in O-ring characteristics resulting from hot immersion testing in the V1066 hydrotreated process solvent are summarized in Table 7. Major differences were found among the seven seal materials investigated. All the seals swelled during the immersion test, but the Kalrez, Viton, and Durafite seals were significantly more stable, dimensionally and volumetrically, than any of the EPDM seals. Durafite was inflexible (rigid) before and after testing, whereas the relative flexibility of the Kalrez and, to a much greater degree, of the Viton decreased. However, the flexibility of the three EPDM O-ring seals increased significantly. The swelling of the Durafite seal was anisotropic, 10% in the thickness (axial) direction but only 1.3% in the radial cross section, the smallest change observed. Swelling in the axial direction would be accommodated by the compression springs in the P1039 pump and, therefore, may not be a disadvantage. The swelling of the elastomer seals appeared to be isotropic, and the volume increase of these seals was found to be directly proportional to their weight gain.

In order of increasing volume change, Kalrez 1050 was lowest followed by Kalrez 4079, Viton, Durafite, Precision EPDM 42679, Parker EPDM E962-85, and Parker EPDM 692-75. The swelling ranged from about 2.6 vol % for the Kalrez 1050 to about 123 vol % for the Parker EPDM 692-75. Of the three EPDM O-ring seals in the study, Precision's 42679 swelled the least, about 81 vol %, and it had the smallest increase in relative flexibility on an absolute basis. However, the percentage increase in relative flexibility was 717%, the largest increase found in the study. The Viton swelled by only 10.6 vol %, but its relative flexibility decreased by 92%.

On the basis of these limited hot *immersion* tests, the DuPont Kalrez 1050 and 4079 are the first and second choices, respectively, for pilot plant service performance tests at Wilsonville. Of the three EPDM

Table 7. Summary of changes in O-ring cross section volume, weight, density, and deflection resulting from hot immersion testing

Identification	Change (%)				
	Cross section	Volume	Weight	Density	Deflection
Durametallic Durafite	+1.3	+13.8	+59.8	+39.0	0
Parker					
EPDM E692-75	+30.2	+122.6	+93.0	-13.3	+218.3
EPDM E962-85	+25.8	+93.7	+78.1	-8.1	+425.2
Precision					
EPDM 42679	+22.2	+80.7	+66.7	-7.8	+717.0
Viton 16609	+3.3	+10.6	+4.0	-6.0	-92.1
Dupont					
Kalrez 1050	+1.4	+2.6	+1.3	-1.9	
Kalrez 4079	+1.8	+4.9	+1.9	-2.8	

compounds in the study, Precision's 42679 is the most promising. It should be stressed that the L'Garde report¹ reached the same conclusions on the basis of their immersion tests. It was not until L'Garde conducted static *simulation* tests [in which the O-rings were constrained while a differential pressure of about 20.7 MPa (3000 psi) was maintained] that the superior performance of the Precision EPDM 42679 was identified. It appears that confinement (by backup rings, on a small gland opening)¹ or compressive stress reduces the rate of swelling of the elastomer, thereby prolonging the service life of the elastomer.

We recommend that O-ring seals be molded or turned to a rectangular cross section (like the Durafite seal) to fill the gland opening, thereby minimizing swelling, and that backup rings be used to reduce the permeation rate. Possibly, the compression springs in Wilsonville's existing Durametallic double seals could be used to obtain adequate sealing performance from the relatively inexpensive Precision EPDM 42679 compound. It is possible that implementation of these recommendations would improve the service performance on many elastomer compounds, including Kalrez 1050.

In-plant evaluation of O-rings was initiated at Wilsonville on March 6, 1985, with the installation of two E962-85 (Parker Seals) O-rings in the P1039 pump. About 13 months (8842 h of total run time) later, the initial set of O-rings continues to perform satisfactorily.

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