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Program: Cover Gas Seals

AEC Task: 11 - FFTF-LMFBR Seal Test Program

Program Manager: G. W. Meyers

Project Manager: J. G. Asquith

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Principal Investigators: W. Kurzeka, R. Oliva, F. Welch, T. Shimazaki

I. PROJECT OBJECTIVES

The objectives of this program are to: (1) conduct static and dynamic tests to demonstrate or determine the mechanical performance of full-size (cross section) FFTF fuel transfer machine and reactor vessel head seals intended for use in a sodium vapor - inert gas environment, (2) demonstrate that these FFTF seals or new seal configurations provide acceptable fission product and cover gas retention capabilities at Clinch River Breeder Reactor Plant (CRBRP) operating environmental conditions other than radiation, and (3) develop improved seals and seal technology for the CRBRP to support the national objective to reduce all atmospheric contaminations to low levels.

The leakage and/or permeation of reactor cover gas into the FFTF head cavity through elastomer seals will be calculated for each seal by examination of head-region penetrations and use of permeation measurements and/or seal permeation measurements generated on this program.

An additional objective will be to conduct a feature test on the IVHM inflatable seals to: (1) verify acceptable operational performance under environmental conditions anticipated in FFTF service, and (2) demonstrate satisfactory long-term operation (160,000 linear feet of travel) for the seals at the anticipated CRBRP rotating plug speed. A feature test to assess the performance of the FFTF Instrument Tree inflatable seals will be conducted.

A special test will be conducted by AI and the Hanford Engineering Development Laboratory (HEDL), wherein the permeation of a set of elastomer seals will be measured by AI and the leakage of a metallic seal measured by HEDL. The test fixtures and gases will be exchanged between the two facilities, and the tests repeated, to compare the measuring techniques of the two laboratories.

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A top shield dip seal parametric test will be conducted to establish the optimum configuration of the liquid sodium dip seal.

II. TECHNICAL PROGRESS DURING FISCAL YEAR 1974

A. DYNAMIC TESTING

Testing is continuing on all phases of the dynamic seal test program to provide meaningful test data on a time schedule consistent with FFTF and CRBRP program requirements. Two types of seal tests are being run in the program: demonstration tests and information tests. The demonstration tests are evaluating the performance of various seal types when tested under conditions representative of those specified for FFTF seal design conditions. Testing is about 6 months in duration for each group of test seals. Information tests are measuring the short-term performance of various seal types under broader ranges of seal application conditions, to determine appropriate new guidelines and/or seal standards for use in the design of current or future reactor systems.

Two types of motion, linear reciprocating and rotary reciprocating (10 rotations between direction changes), are being studied (Figures 47 and 48). Each region between pairs of seals contains a different concentration of argon, hydrogen, krypton, and xenon. At weekly intervals, samples of gas are removed and analyzed using a trace gas analyzer employing a gas chromatographic technique. The concentrations of hydrogen, krypton, and xenon found in each region (argon is not measured since it is the common gas throughout the fixture) enable the permeation and leakage of a particular gas specie through a specific seal to be determined. Table 22 shows a typical tabulation of leakage data for Reciprocating Demonstration Test 2 containing two different vendors' Buna N (Nitrile) seals.

The initial calculations, obtained using the vendor's recommended method for calibrating the trace gas analyzer, were significantly lower than would be expected if the leakage were calculated using the measured permeability values for the particular elastomer (see Section III-C). Three bottles of certified calibrating gas were purchased, each containing a different concentration of hydrogen, krypton, and xenon in argon. Recalibrating the trace gas analyzer produced markedly different calibration curves which, in turn, gave higher leakage values.

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Figure 47. Reciprocating Seal Test Fixture

9006-4048A

Figure 48. Rotary Seal Test Fixture

TABLE 22
ROTATING DEMONSTRATION TEST NO. 3

Seal Gland		Seal Manufacturer and Type	Compression (%)				
A B C D		Minnesota Rubber Quad Rings, Buna N, Compound 366Y	10				
Test Temperature = 150 °F				Shaft Finish: 8 rms			
Lubricant: DC-55M				Gland Finish: 32 rms			
Elapsed Time (hr)	Actual Running Time (hr)	No. of Cycles	Total Travel (ft)	Cavity Trace Gas Measurements			
				Total cc Gas			
				A	B	C	
				Kr	Xe	H ₂	
172	36	2533	1325	0.007	0.020	0.065	
316	73	5080	2660	0.031	0.042	0.094	
556	131	9180	4800	0.039	0.054	0.125	
700	162	11350	5950	0.049	0.095	0.148	
1060	247	17280	9050	0.055	0.079	0.182	
1492	390	27335	14300	0.084	0.106	0.255	

The leakage calculations for all the test fixtures have been programmed for computer handling which permit calculation refinements and corrections to be made which are too tedious and time-consuming for manual handling.

Program redirection was given by HEDL/ARD in April to include urethane seals in the test program. Concurrently, the half of the information test matrix which included the second lubricant tests was deleted. Only selected comparison tests will be made with the second lubricant, WSX 87788.

Individual tests which were completed or initiated this fiscal year are discussed in the following sections.

1. Reciprocating Seal Tests

- a. Demonstration Tests

Table 23 summarizes the characteristics and performance of all reciprocating demonstration tests completed or currently in test. None of these tests developed seal leaks even though the seals operated far more than the 2100 ft required by the Westinghouse document, WD TRS 1.25, except Test 3 which developed a seal leak very shortly after startup. When this test was disassembled, it was determined that shaft misalignment had caused one of the T-seals to wear extensively.

The test was restarted with a new set of seals (Test 3 Rerun) and a more careful alignment of the shaft and housing. Figures 49 and 50 are close-up views of a typical Buna N T seal and Q seal, respectively, showing the absence of wear. The success of the rerun vs the initial test points up the importance of proper alignment of the reciprocating shaft to prevent unnecessary wear and the seal leakage which accompanies the wear.

Figure 51 shows a comparable photograph from Test 4 indicating that the EPR seals under similar test conditions did not show noticeable wear.

Demonstration Tests 5 and 6 are currently in progress. These tests are a part of the revised test program.

TABLE 23
RECIPROCATING DEMONSTRATION SEAL TEST

Test	Seals	Compression (%)	Shaft Finish (rms)	Temperature (°F)	Lubricant	Seal Travel (ft)	Comments
1	T seals (3) EPR Greene Tweed	10	8	150	DC55M	8151 (6120 cycles)	Long-term test, observed leakage primarily due to permeation, no gross changes in sealing characteristics noted. Test stopped because of leaky bellows.
	Q rings (3) EPR Parker Seal	10	8	150	DC55M	8151 (6120 cycles)	
2	Quad rings (3) Buna N Minnesota Rubber	10	8	150	DC55M	5108 (3830 cycles)	No significant leakage observed
	T seals (3) Buna N Greene Tweed	10	8	150	DC55M	5108 (3830 cycles)	
3 Rerun	Q rings (3) Buna N Parker Seal	10	8	150	DC55M	6210 (4660 cycles)	No significant changes or leakage noted
	T seals (3) Buna N Parker Seal	10	8	150	DC55M	6210 (4660 cycles)	
4	Q rings (3) EPR Minnesota Rubber	10	8	150	DC55M	6210 (4660 cycles)	No significant changes or leakage noted
	T seals (3) EPR Parker Seal	10	8	150	DC55M	6210 (4660 cycles)	
5	T seals (3) Urethane Parker Seal	10	8	100	DC55M		In test
	T seals (3) Buna N Greene Tweed	10	8	100	DC55M		
6	T seals (3) Urethane Parker Seal	10	8	150	WSX8788		In test
	T seals (3) Buna N Greene Tweed	10	8	150	WSX8788

Figure 49. Parker Seal T-Seal Removed from Reciprocating Demonstration Test 3 Rerun
After Six Months of Testing

9006-40182

Figure 50. Parker Seal Q-Ring Removed from Reciprocating Demonstration Test 3 Rerun
After Six Months of Testing

nm540184

Figure 51. Minnesota Rubber Quad-Ring Removed from Reciprocating Demonstration
Test 4 After Six Months of Testing

9006-40185

b. Information Tests

Table 24 summarizes the reciprocating information seal tests conducted during the year. Reciprocating Information Test 6^{*} was the first of the tests on silicone rubber seals and, as noted in Table 24, gross leakage occurred at the 5% compression end of the fixture, but only after the seals had far exceeded the requirements of the WDTRS 1.25 document which defines the seal operating conditions and test requirements. Only minimal wear was observed, although some seal debris was found (Figure 52). Shown in Figure 52 is the silicone rubber closure O-ring which suffered about 43% compression set during the 486-hr testing at 300°F. Figures 53 and 54 show the test shafts which show only a thin layer of black residue and longitudinal rub marks.

During the period that Reciprocating Information Test 6 was running, the trace gas analyzer was not operable. This required that the test be rerun.[†] During the assembly of the shaft into the housing containing the seals, a thin sliver of silicone rubber was sheared from a T-seal (Figure 55), even though the shaft was tapered to provide a lead-in. The fixture was disassembled and a new seal installed. A small amount of additional lubricant was placed on the T-seal, and the assembly was made without damage to the seal. The damage to the silicone rubber seal emphasizes the care which must be taken when using silicone rubber seals which are known to be "fragile" or "tender" and easily damaged.

The seals were given a total of 6550 cycles during a 200-hr period without observable pressure changes in the regions between the 20% compression seal pairs, although pressure changes had been noted in the regions between the 5% compression seals after 4920 cycles. When the fixture was disassembled two discrepancies were found:

- 1) Two silicone rubber O-rings had been accidentally installed in the 5% compression end of the fixture instead of two silicone rubber Quad-rings. It was further found that one of the O-rings had suffered a "spiral failure" as noted in Figure 56.[§] This error dramatically

^{*}Information Tests 1 through 5 were reported in the previous annual report, AI-AEC-13110.

[†]A second TGA has since been ordered and put into operation.

[§]The O-ring seal is known for its susceptibility to spiral failure in a reciprocating application where the seal rolls in alternating directions.

TABLE 24
RECIPROCATING INFORMATION SEAL TEST
(Sheet 1 of 2)

Test	Seals	Com pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
6	T seal (1) Silicone Parker Seal	5	4	300	DC55M	6000 (4500 cycles)	Gross leakage of 5% com- pression seals indicated at ~5250 ft of travel (two times the required WDTRS life, but less than the Test Plan lifetime). Test termi- nated. No significant leak- age of 20% seals.
	Quad ring (2) Silicone Minnesota Rubber						
	Q ring (1) Silicone Parker seal	20	4				
	T rings (2) Silicone Greene Tweed						
6 Rerun	T seal (1) Silicone Parker Seal	5	4	300	DC55M	6550 (4500 cycles)	Gross leakage of 5% com- pression seals shown after 4920 cycles (6550 ft) of travel. No leakage of 20% compression seals.
	Quad rings (2) Silicone Minnesota Rubber						
	Q ring (1) Silicone Parker Seal	20	4				
	T rings (2) Silicone Greene Tweed						
7	T seals (2) Buna N Greene Tweed	5	16	150	DC55M	8590 (6460 cycles)	Test completed. No gross leakage observed. Seals appeared in good shape.
	Q ring (1) Buna N Parker Seal						
	T seal (1) Buna N Parker Seal	20	16				
	Quad rings (2) Buna N Minnesota Rubber						

TABLE 24
RECIPROCATING INFORMATION SEAL TEST
(Sheet 2 of 2)

Test	Seals	Com- pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
8	T seal (1), EPR Parker Seal	5	16	150	DC55M	10,850 (8153 cycles)	Test completed. No leak- age observed, except at end bellows.
	Quad rings (2), EPR Minnesota Rubber	5	16				
	Q ring (1), EPR Parker Seal	20	16				
	T rings (2), EPR Greene Tweed	20	16				
9	T seal (1) Urethane No. 379 Greene Tweed	5	16	100	DC55M	13,300 (10,000 cycles)	Required number of cycles completed; no leakage observed.
	Q rings (2) Urethane No. P648 Parker Seal	5	16				
	Quad ring (1) Urethane No. 522FX Minnesota Rubber	20	16				
	T seals (2) Urethane No. P648 Parker Seal	20	16				
10	T seal (1) Silicone No. 407 Greene Tweed	5	16	300	DC55M	4900 (3676 cycles)	Test stopped gross leakage in 5% com- pression seals.
	Q rings (2) Silicone No. S684 Parker Seal	5	16				
	Quad ring (1) Silicone No. 71417 Minnesota Rubber	20	16				
	T seals (2) Silicone No. S684 Parker Seal	20	16

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Figure 52. End Housing of Reciprocating Information Test 6

Figure 53. 5% Compression End of Shaft From Reciprocating Information Test 6

9006-4073

Figure 54. 20% Compression End of Shaft From Reciprocating Information Test 6

9006-4074

Figure 55. T-Seal Failure During Assembly

9006-4089

9006-4094

Figure 56. O-Ring Failure During Reciprocating Information Test 6

pointed out the need for careful seal control, and the potentially drastic result of the installation of the wrong type of seal. An Operating Procedure (OP 707-810-002) was prepared to establish seal control for future tests.

- 2) Examination of the test fixture revealed that the seal debris had clogged the gas ports on the 20% compression end of the fixture, and prevented the gas pressure changes on the manometer which would have indicated gross leakage. The combination of lubricant, seal debris, and a 300°F temperature produced a dense plug which required several psig pressure to "blow out."

This plugging action had not been observed previously in the tests, and emphasizes the need in the FFTF and the LMFBR to provide adequately sized and located gas ports between dynamic seal pairs to minimize plugging. It also points out a need for detecting and removing a gas port plug, if it occurs, since the gas ports are necessary to provide a positive pressure of inert gas between seals to minimize the outward leakage of radioactive gases in the event of seal failure.

Test 7, containing Buna N seals, as noted in Table 24, completed ~390 hours of testing (8590 ft) with no evidence of gross leakage. The system was disassembled, and the seals were removed. Examination revealed all seals to be in good condition.

Test 8 completed 10,850 ft of travel with no evidence of seal leakage, although a leak in the bellows was noted near the end of the test (Table 24). This test was continued beyond the normal scheduled cycling to obtain additional gas chromatographic data. Examination of the seals revealed all seals to be in good condition.

Reciprocating Test 9 containing urethane seals, (Table 24) completed ~13,300 ft with no evidence of leakage. (Note: Only 2100 ft are required by the WDTRS 1.25 document, but the test was extended to obtain longer-term leakage data.)

Reciprocating Test 10, containing silicone seals as noted in Table 24 was run for 3676 cycles (4900 ft) before the test was stopped when gross leakage occurred on the 5% compression seals. It should be noted that the leakage did not occur in this test, or in any other reciprocating information test until the specified travel had been exceeded by at least a factor of two.

2. Rotating Seal Tests

a. Demonstration Tests

Table 25 summarizes the rotating demonstration seal tests completed this year. No tests have indicated gross seal leakage, even though the seal travel exceeded the 28,000 ft requirement of the WDTRS 1.25 document. Post-test examination of the seals from these tests have shown only minimal wear.

b. Information Tests

Table 26 summarizes the 18 rotating information seal tests conducted during the year. Tests 6, 7, 11, and 12 of Buna N seals completed the test sequence without evidence of leakage at either 5% or 20% compression.

Tests 1, 5, and 8 of EPR completed the run without evidence of gross leakage, but Test 9, at 20% compression, was stopped at 10,000 ft of travel because of high friction forces. No evidence of wear was observed on any of the tests, although for the No. 9 seals high friction forces were recorded on the transducer. In this test, a torque wrench was attached to the shaft and a breakaway force of 110 in.-lb and a running force of 190 in.-lb were measured, which are equivalent to ~ 4.4 and 7.6 in.-lb/in. of seal, respectively. The test was continued to obtain additional gas chromatographic data.

Tests 10, 13, and 14 on silicone rubber seals had indications of leakage, but because of the high permeability of silicone rubber, it is difficult to distinguish between a very small leak and permeation. The amount of visible wear and wear debris was minimal on all three tests. The nick noted on Figure 57 by the arrows was caused during disassembly. Silicone rubber is generally not tough and durable, and is easily damaged.

TABLE 25
ROTATING SEAL DEMONSTRATION TESTS

Test	Seals	Com- pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
1	Q rings (4), EPR Parker Seal	10	8	150	DC55M	31,060 (59,395 cycles)	Test completed. No significant leakage observed.
2	O rings (4), EPR Minnesota Rubber	10	8	150	DC55M	31,060 (59,395 cycles)	Test completed. No significant leakage observed until after re- quired number of cycles were completed.
3	Quad rings (4), Buna N Minnesota Rubber	10	8	150	DC55M	31,380 (60,000 cycles)	Test completed. No indication of gross leakage.
4	O rings (4) Buna N Parker Seal	10	8	150	DC55M	31,380 (60,000 cycles)	Test completed. No indication of gross leakage.
5	Q rings (4) Buna N Parker Seal	10	8	150	DC55M	32,070 (61,325 cycles)	Test completed. No indication of leakage.
6	O rings (4) Buna N Minnesota Rubber	10	8	150	DC55M	32,070 (61,325 cycles)	Test completed. No indication of leakage.
7	Quad rings (3) EPR Minnesota Rubber*	10	8	150	DC55M	18,125 (34,653 cycles)	Test in progress; no indication of leakage.
8	O rings (3) EPR Parker Seal*	10	8	150	DC55M	18,125 (34,653)	Test in progress; no indication of leakage.
9	O rings (3) Urethane Parker Seal	10	8	100	DC55M	1,790 (3,423 cycles)	Test in progress; no indication of leakage.
10	O rings (3) Urethane Parker Seal	10	8	150	DC55M	1,790 (3,423 cycles)	Test in progress; no indication of leakage.

*Parker Seal Buna N (N741) outer seal to reduce end losses.

TABLE 26
ROTATING SEAL INFORMATION TESTS
(Sheet 1 of 3)

Test	Seals	Com- pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
1	O rings (2) EPR Minnesota Rubber	5	4	150	DC55M	40,605 (77,640 cycles)	No significant leakage ob- served. Test stopped. Minimal seal wear observed.
	O rings (2) EPR Parker Seal						
5	O rings (2) EPR Parker Seal Co.	20	4	150	DC55M	40,705 (77,830 cycles)	No significant leakage ob- served. Test stopped. Minimal seal wear observed.
	Quad-ring (2) EPR Minnesota Rubber						
6	Quad rings (2) Buna N Minnesota Rubber	5	4	150	DC55M	32,580 (63,300 cycles)	Test terminated. No indication of gross leakage.
	O rings (2) Buna N Parker Seal						
7	Q rings (2) Buna N Parker Seal	20	4	150	DC 55M	32,580 (62,300 cycles)	Test terminated. No indication of gross leakage.
	O rings (2) Buna N Minnesota Rubber						
8	Quad ring (1) EPR Minnesota Rubber	5	16	150	DC55M	27,680 (52,930 cycles)	No indication of gross leakage.
	O rings (2) EPR Parker Seal						
9	Q rings (1) EPR Parker Seal	20	16	150	DC55M	10,000 (19,100 cycles)	No indication of gross leakage. High friction forces. Test stopped.
	O rings (2) EPR Minnesota Rubber						

TABLE 26
ROTATING SEAL INFORMATION TEST
(Sheet 2 of 3)

Test	Seals	Com- pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
10	O rings (1) Silicone Parker Seal	5	4	300	DC55M	27,680 (52,930 cycles)	Suspected leak.
	Quad rings (2) Silicone Minnesota Rubber						
11	O rings (1) Buna N Minnesota Rubber	5	16	150	DC55M	31,304 (59,855 cycles)	Test terminated. No indication of gross leakage.
	Q rings (2) Buna N Parker Seal						
12	O ring (1) Buna N Parker Seal	20	16	150	DC55M	31,304 (59,855 cycles)	Test terminated. No indication of gross leakage.
	Quad rings (2) Buna N Minnesota Rubber						
13	Q ring (1) Silicone Parker Seal	20	4	300	DC55M	27,680 (52,930 cycles)	Suspected leak.
	O ring (2) Silicone Minnesota Rubber						
14	O ring (1) Silicone Minnesota Rubber	5	16	300	DC55M	32,150 (61,484 cycles)	Test stopped, leakage noted across seals.
	Q rings (2) Silicone Parker Seal						
15	Quad ring (1) Silicone Minnesota Rubber	20	16	300	DC55M		Test completed.
	O rings (2) Silicone Parker Seal						

TABLE 26
ROTATING SEAL INFORMATION TEST
(Sheet 3 of 3)

Test	Seals	Com- pression (%)	Shaft Finish (rms)	Temper- ature (° F)	Lubricant	Seal Travel (ft)	Comments
16	O ring (1) Urethane Parker Seal	5	4	100	DC55M		Test completed.
	Quad rings (2) Urethane Minnesota Rubber						
17	Q ring (1) Urethane Parker Seal	20	4	100	DC55M	16,420 (31,400 cycles)	Test stopped. Friction forces too high.
	O rings (2) Urethane Minnesota Rubber						
18	O ring (1) Urethane Minnesota Rubber	5	16	100	DC55M	53,535 (108,100 cycles)	Test completed. No leakage observed.
	Q rings (2) Urethane Parker Seal						
19	Quad ring (1) Urethane Minnesota Rubber	20	16	100	DC55M	12,400 (23,680 cycles)	Test rotation stopped. High torque forces.
	O rings (2) Urethane Parker Seal						
20	O ring (1) Urethane Minnesota Rubber	5	4	100	WSX-8788	56,535 (108,100 cycles)	Test completed.
	Q rings (2) Urethane Parker Seal						
21	Quad ring (1) Urethane Minnesota Rubber	20	4	100	WSX-8788	56,535 (108,100 cycles)	Test completed.
	O rings (2) Urethane Parker Seal

Figure 57. Rotary Seal Information Test 10, Minnesota Rubber Co.
Silicone Quad Ring Nicked During Assembly

9006-40147A

Figure 58. Rotary Information Test 17,
Urethane Quad Ring, Showing Wear

9006-40167A

Tests 17 and 19 on urethane rubber were stopped short of the test cycle because of high friction forces caused by the 20% compression seals. One of the seals removed from Test 17 showed significant wear. Figure 58 is a close-up view of the seal (outer containment seal, urethane Q-ring). This seal apparently rotated in the groove, where there is a minimum of lubrication, and subsequently produced a high torque. This wear was not observed in any of the other seals.

Tests 16, 18, 20, and 21 completed over 56,000 ft of travel (twice the WDTRS 1.25 requirement) without evidence of wear or leakage. The latter two tests (20 and 21) were run using the WSX-8788 grease, an ester-type lubricant which is discussed in a later section of this report.

c. Conclusions

In general, it is concluded from these tests that the 5% compression is too small and may lead to seal leakage; the 20% compression is too high and may produce high friction forces. All seals (Buna N, EPR, and urethane) performed well as materials, and exhibited problems only from the geometrical factor of compression. The silicone seals generally were the worst performers, exhibiting higher leakage and wear.

When the performance of all seals are compared (Demonstration and Information), it is concluded that the 10% compression is the optimum choice. Since no significant differences were noted between the 4-, 8-, and 16-rms finish from the aspects of wear and gross leakage, the 8-rms shaft finish was selected as the optimum finish for the rubbing metal surface.

3. Leakage Calculations

The amounts of H_2 , Kr, and Xe gas permeating through or leaking around the seals during test are measured with a highly sensitive gas chromatograph capable of detecting from <1 ppm to nearly 1% of the trace gas. These gas concentrations then are processed with a computer program which converts the concentrations to seal leakage using the appropriate sample chamber volume, gas temperature, pressure, seal length, differential pressure of the trace gas (driving force), etc. Initially no corrections were made for the gases leaking past the outer or containment seal to the atmosphere. This amount is small for

seals of low permeability, but significant for seals of higher permeability. The computer program also corrects for these system losses. The majority of the data have been processed and are noted in Tables 27-34 which present the measured leakage data expressed in std cc/sec/linear in. of seal/atm ΔP for each of the test gases (H_2 , Kr, and Xe). The tables also present a second leakage value — one which was calculated from the elastomer permeability data (described in a later section), the seal geometry, % compression, and test temperatures for a simple static, O-ring geometry. These calculated values provide useful comparison information. Seals of differing geometry may be expected to exhibit consistently higher, or consistently lower, leakages than an O-ring seal. Likewise, dynamic seals should be expected to exhibit higher leakages than static seals.

Reviewing these data for the dynamic seal leakages tabulated in this section and the static seal leakages tabulated later in the report, several conclusions can be drawn even though the calculations are not entirely complete at this time:

- 1) The data from the reciprocating seal tests are generally erratic. The data from the demonstration tests agree much better with the estimated values than do the information test data. This is not unexpected, since the motion of the shaft backwards and forwards through the seals presents an ever-changing seal geometry conducive to leakage and unstable behavior.
- 2) The data from the rotary seal tests and the static seal tests generally agree quite well with the estimated values.
- 3) The Buna N and EPR rubbers behave predictably, and generally the leakage values agree well with the predicted values, whereas the leak rate through the silicone seals is so high at the 300°F test temperature that it is difficult for the measuring technique to follow the rapid changes in gas concentration.

TABLE 27
RECIPROCATING BUNA N SEALS
L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage					
							L _{H2}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
T	Buna N*	GT	ReD-2	10	8	DC55M	91	90	23	23	—	16
T	Buna N*	PS	ReD-3	10	8	DC55M		78		20		24
T	Buna N†	GT	ReD-5	10	8	DC55M		33		3		3
Quad	Buna N*	MR	ReD-2	10	8	DC55M	160	114	61	33	—	40
Q	Buna N*	PS	ReD-3	10	8	DC55M		78		20		24
T	Buna N*	GT	ReD-6	10	8	WSX8788		90		13		16
T	Buna N*	PS	ReI-5	5	4	DC55M		90		23		28
T	Buna N*	GT	ReI-4	20	4	DC55M	—	70	13	10	—	12
T	Buna N*	GT	ReI-7	5	16	DC55M	—	120	47	16	—	19
T	Buna N*	PS	ReI-7	20	16	DC55M	400	62	—	16	—	20
Quad	Buna N*	MR	ReI-5	5	4	DC55M		131		38		46
Quad	Buna N*	MR	ReI-4/7	20	4/16	DC55M	1,000	90	37	26	—	31
Q	Buna N*	PS	ReI-7	5	16	DC55M	435	90	—	23	—	28
Q	Buna N*	PS	—	20	4	DC55M		62		16		20

GT = Greene Tweed

PS = Parker Seal

MR = Minnesota Rubber

Re = Reciprocating

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr $\times 10^{-8}$ (L_{H2} and L_{Xe} similar units)

*150° F

†100° F

TABLE 28
RECIPROCATING EPR SEALS
L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage					
							LH ₂		LK _r		LX _e	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
T	EPR*	GT	ReD-1	10	8	DC55M	620	224	90	130	341	105
T	EPR	PS	ReD-4	10	8	DC55M		313		130		347
Quad	EPR	MR	ReD-4	10	8	DC55M		420		170		279
Q	EPR	PS	ReD-1	10	8	DC55M	954	313	118	118	445	247
T	EPR	GT	ReI-4	5	4	DC55M	—	242	140	140	138	113
T	EPR	PS	ReI-5	20	4	DC55M		231		96		182
T	EPR	PS	ReI-8	5	16	DC55M	—	362	30	150	—	285
T	EPR	GT	ReI-8	20	16	DC55M	—	165	—	96	24	78
Quad	EPR	MR	ReI-8	5	16	DC55M	—	469	—	190	45	312
Quad	EPR	MR	ReI-5	20	4	DC55M		321		130		213
Q	EPR	PS	ReI-4	5	4	DC55M	1,100	362	—	150	—	285
Q	EPR	PS	ReI-8	20	16	DC55M	517	231	—	96	—	182

GT = Greene Tweed

PS = Parker Seal

MR = Minnesota Rubber

Re = Reciprocating

I = Information

D = Demonstration

LK_r = Std cc/sec/linear in. of seal/atm ΔP of Kr x 10⁻⁸ (LH₂ and LX_e similar units)

*150° F

TABLE 29
RECIPROCATING URETHANE SEALS
L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage					
							L _{H2}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
T	Urethane*	PS	ReD-5	10	8	DC55M		15		2		3
T	Urethane†	PS	ReD-6	10	9	WSX8788		51		16		17
T	Urethane*	PS	ReI-1	5	4	DC55M		19		2.5		4
T	Urethane*	GT	ReI-1	20	4	DC55M		27		4.5		4
T	Urethane*	GT	ReI-9	5	16	DC55M	—	42	100	7	—	6.5
T	Urethane*	PS	ReI-9	20	16	DC55M	—	11	135	1.5	88	2
Quad	Urethane*	MR	ReI-1	5	4	DC55M		45		8		7.5
Q	Urethane*	PS	ReI-1	20	4	DC55M		11		1.5		2
Q	Urethane*	PS	ReI-9	5	16	DC55M	196	19	—	2.5	79	4
Quad	Urethane*	MR	ReI-9	20	16	DC55M	173	28	—	5	—	5

GT = Greene Tweed

PS = Parker Seal

MR = Minnesota Rubber

Re = Reciprocating

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr x 10⁻⁸ (L_{H2} and L_{Xe} similar units)

*100° F

†150° F

TABLE 30
RECIPROCATING SILICONE SEALS
L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage					
							L _{H2}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
T	Silicone*	PS	ReI-6	5	4	DC55M		13,055		6,800		7,685
T	Silicone*	GT	ReI-6	20	4	DC55M		16,500		3,100		26,000
T	Silicone*	GT	ReI-10	5	16	DC55M		23,800		4,500		37,600
T	Silicone*	PS	ReI-10	20	16	DC55M		8,830		4,600		5,200
Quad	Silicone*	MR	ReI-6	5	4	DC55M		22,140		9,000		6,930
Q	Silicone*	PS	ReI-6	20	4	DC55M		8,830		4,600		5,200
Q	Silicone*	PS	ReI-10	5	16	DC55M		13,055		6,800		7,685
Quad	Silicone*	MR	ReI-10	20	16	DC55M		14,760		6,000		4,620

GT = Green Tweed

PS = Parker Seal

MR = Minnesota Rubber

Re = Reciprocating

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr $\times 10^{-8}$ (L_{H2} and L_{Xe} similar units)

*300° F

TABLE 31
ROTATING BUNA N SEAL, L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer *	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage [†]					
							L _{H₂}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
O	Buna N	PS	RoD-4	10	8	DC55M	41	78	26	20	35	24
O	Buna N	MR	RoD-6	10	8	DC55M	36	114	31	33	36	40
Quad	Buna N	MR	RoD-3	10	8	DC55M	112	114	29	33	41	40
O	Buna N	PS	RoD-5	10	8	DC55M	46	78	30	20	30	24
O	Buna N	PS	RoI-6	5	4	DC55M	-	90	51	23	25	28
O	Buna N	MR	RoI-7	20	4	DC55M	148	90	-	26	33	31
O	Buna N	MR	RoI-11	5	16	DC55M	-	131	8	38	-	46
O	Buna N	PS	RoI-12	20	16	DC55M	-	62	33	16	-	20
Quad	Buna N	MR	RoI-6	5	4	DC55M	91	131	-	38	-	46
Q	Buna N	PS	RoI-7	20	4	DC55M	-	62	19	16	-	20
Q	Buna N	PS	RoI-11	5	16	DC55M	122	90	-	23	24	28
Quad	Buna N	MR	RoI-12	20	16	DC55M	93	90	-	26	24	31

PS = Parker Seal

MR = Minnesota Rubber

Ro = Rotary

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr $\times 10^{-8}$ (L_{H₂} and L_{Xe} similar units)
*150°F

[†]Listed values corrected for permeation leakage through outer fixture seal

TABLE 32
ROTATING EPR SEALS, L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer*	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage [†]					
							L _{H₂}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
O	EPR	MR	RoD-2	10	8	DC55M	79	420	460	170	246	279
O	EPR	PS	RoD-8	10	8	DC55M	-	313	-	130	-	247
Quad	EPR	MR	RoD-7	10	8	DC55M	-	420	-	170	-	279
Q	EPR	PS	RoD-1	10	8	DC55M	79	313	136	130	341	247
O	EPR	MR	RoI-1	5	4	DC55M	-	469	80	190	75	312
O	EPR	PS	RoI-5	20	4	DC55M	151	231	-	96	-	182
O	EPR	PS	RoI-8	5	16	DC55M	126	362	-	150	56	285
O	EPR	MR	RoI-9	20	16	DC55M	138	321	-	130	50	213
Q	EPR	PS	RoI-1	5	4	DC55M	258	362	-	150	-	285
Quad	EPR	MR	RoI-5	20	4	DC55M	-	321	83	130	64	213
Quad	EPR	MR	RoI-8	5	16	DC55M	-	469	101	190	-	312
Q	EPR	PS	RoI-9	20	16	DC55M	-	231	70	96	-	182

PS = Parker Seal

MR = Minnesota Rubber

Ro = Rotary

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr × 10⁻⁸ (L_{H₂} and L_{Xe} similar units)
*150°F

† Listed values corrected for permeation leakage through outer fixture seal

TABLE 33
ROTATING URETHANE SEALS
L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage§					
							L _{H2}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
O	Urethane*	PS	RoD-9	10	8	DC55M	-	15	-	2	-	3
O	Urethane†	PS	RoD-10	10	8	DC55M	-	51	-	16	-	17
O	Urethane*	PS	RoI-16	5	4	DC55M	-	19	-	2.5	-	4
O	Urethane*	MR	RoI-17	20	4	DC55M	-	28	-	5	-	5
O	Urethane*	MR	RoI-18	5	16	DC55M	-	45	-	8	-	7.5
O	Urethane*	PS	RoI-19	20	16	DC55M	-	11	-	1.5	-	2
Quad	Urethane*	MR	RoI-16	5	4	DC55M	-	45	-	8	-	7.5
Q	Urethane*	PS	RoI-17	20	4	DC55M	-	11	-	1.5	-	2
Q	Urethane*	PS	RoI-18	5	16	DC55M	-	19	-	2.5	-	4
Quad	Urethane*	MR	RoI-19	20	16	DC55M	-	28	-	5	-	5
O	Urethane*	MR	RoI-20	5	4	WSX8788		45		8		7.5
O	Urethane*	PS	RoI-21	20	4	WSX8788		11		1.5		2
Q	Urethane*	PS	RoI-20	5	4	WSX8788		19		2.5		4
Quad	Urethane*	MR	RoI-21	20	4	WSX8788		28		5		5

PS = Parker Seal

MR = Minnesota Rubber

Ro = Rotary

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr $\times 10^{-8}$ (L_{H2} and L_{Xe} similar units)

*100° F

†150° F

§ Listed values corrected for permeation leakage through outer fixture seal

TABLE 34
ROTATING SILICONE SEALS, L VALUES FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer *	Supplier	Test Type	Com- pression (%)	Finish (rms)	Lubricant	Leakage †					
							L _{H₂}		L _{Kr}		L _{Xe}	
							Meas.	Calc.	Meas.	Calc.	Meas.	Calc.
O	Silicone	PS	RoI-10	5	4	DC55M	-	13,055	644	6,800	-	7,685
O	Silicone	MR	RoI-13	20	4	DC55M	2,887	14,760	-	6,000	(26)	4,620
O	Silicone	MR	RoI-14	5	16	DC55M	-	22,140	-	9,000	-	6,930
O	Silicone	PS	RoI-15	20	16	DC55M	1,705	8,830	-	4,600	92	5,200
Quad	Silicone	MR	RoI-10	5	4	DC55M	325	22,140	-	9,000	100	6,930
Q	Silicone	PS	RoI-13	20	4	DC55M	-	8,830	481	4,600	-	5,200
Q	Silicone	PS	RoI-14	5	16	DC55M	-	13,055	-	6,800	-	7,685
Quad	Silicone	MR	RoI-15	20	16	DC55M	-	14,760	418	6,000	-	4,620

PS = Parker Seal

MR = Minnesota Rubber

Ro = Rotary

I = Information

D = Demonstration

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr x 10⁻⁸ (L_{H₂} & L_{Xe} similar units)

*300°F

† Listed values corrected for permeation leakage through outer fixture seal

4. Lubricant Evaluation

a. Friction Studies

The program for evaluating candidate lubricants was conducted to select a second lubricant for the seal test program which had been started with the DC55M grease as the primary lubricant. Friction test data obtained on the lubricant evaluation fixture (Figure 59) using Parker Buna N O-rings are shown graphically in Figure 60. Each test was run initially at room ambient, and then heated to 150°F. Of the five lubricants tested (some lubricants were dropped during earlier screening tests), only DC55M, WSX-8788, and SWS-295 exhibited low friction forces.

Similar friction tests were run at ~220°F on Parker Silicone O-rings with the WSX-8788 and the DC55M lubricants, as shown in Figure 61. Examination did not reveal any significant wear of the O-ring. The fixture was reassembled, using the same O-ring as was run with DC55M earlier. The seal was greased, and the test run until failure, as shown on Figure 62, at 99,161 ft of travel. This second portion of testing was run with the seal heated to 300°F.

Three additional friction tests were conducted on urethane, silicone, and EPR elastomer O-rings, lubricated with one of several greases, as shown in Figures 63 through 65. The urethane seals showed low uniform friction forces for both DC55M and WSX-8788 greases.

The Stauffer 295, silicone-base grease (now dropped from the evaluation program) produced high forces with a silicone O-ring, and sheared the safety linkage which protects the transducer. Initially, DC-11 grease on the silicone O-ring produced high forces but these forces dropped off to a low, but variable, value.

A small amount of WSX-8788 was removed from one of the thermal stability tests (see following section), and was used to lubricate a silicone O-ring, to determine any change in lubricity. As shown in Figure 64, the pre-conditioned grease showed about 1-lb force/linear in. of seal, which was the same as the friction force of the as-received material.

Figure 66 shows a bar chart summary of all friction tests run. The tests with Buna N elastomers were run initially, and resulted in the elimination of

a. Fixture

9006-40124A

b. Fixture Installed on Lathe

9006-40123A

Figure 59. Lubricant Evaluation Fixture

73-025-56-56A

Friction 60. Friction Force vs Lubricant - Buna N O-Ring Friction Test,
Run on Parker Buna N O-Rings (Compound N741 Size 345)
at 10% Compression and 48 rpm Continuous at 150 °F

9006-40132

Figure 61. Maximum Friction Force vs Lubricant-on Silicone O Rings
(Temperature = 220 °F, Speed = 48 rpm on 4.0 in. -diameter O Ring)

9006-40133

Figure 62. Friction Force vs Travel – Dow Corning DC55M on Parker Silicone (Compound S684, Size 345)

9006-40168

Figure 63. Friction Force vs Travel – Parker Urethane O Rings
(Compound P648) at 100 °F

9006-40169

Figure 64. Friction Force vs Travel — Parker Silicone O Rings
(Compound S684)

9006-40170

Figure 65. Friction Force vs Travel — Parker EPR O Rings

74-M14-19-21

Figure 66. Friction Force vs Material Elastomer – Lubricant Combination as a Function of Temperature and Distance

the two greases, SWS290 and Super-O-Lube, whose friction force exceeded the limit of the transducer. Tests conducted on silicone elastomers at 220°F eliminated the SWS-295 grease. Tests on EPR at 100°F showed excessive friction for the DC11, and long-term tests on silicone at 300°F showed excessive friction for the reference DC55M grease. The EXXON WSX-8788, a polyester-base grease, showed the best overall results, and was selected as the alternate lubricant in the test program. It is recommended specifically for the lubrication of the silicone elastomer seals.

b. Thermal Stability Tests

The thermal stability of candidate greases was tested by heating weighed specimens of grease in an air furnace for long periods of time. Greases were periodically removed and inspected for weight loss and change in lubricity. Table 35 summarizes test data obtained to date. Tests on all greases except DC55M, DC11, and WSX8788 have been discontinued due to high weight loss, loss of lubricity, or poor performance in the friction studies. While friction tests on DC-11 resulted in high friction, thermal stability tests are continuing as this grease has been selected by Westinghouse as the reference elastomer seal grease for FFTF.

Visual examination of the 400°F specimens after 500 hours shows WSX8788 has the tendency to evaporate the low boiling point esters, while the DC55M separated into a pool of oil probably the base DC51O silicone oil, and the heavier oil and thickner portions of the grease. No change was apparent to the DC11. After 3000 hours both the WSX-8788 and DC55M were totally solidified and the DC11 was firm and gummy. At 350°F, the WSX8788 begins to lose lubricity after about 3000 hours and is quite stiff after 6000 hours. The DC55M has a firm skin, but retains greater lubricity longer than the WSX8788. However, its useful life is nearly reached after 6000 hours. No DC11 specimen yet has exposure in excess of 3000 hours at 350°F for comparison. While the 350 and 400°F temperatures exceed the expected use temperature, they provide insight into the behavior of the compounds.

At 300°F, the WSX8788 appears to have slightly better retention of lubricity and less weight loss than the DC55M.

TABLE 35
LUBRICANT THERMAL STABILITY (WEIGHT LOSS) TEST

Temperature (°F)	Lubricant	Total Weight Loss (%)														Condition at Last Weight Loss Measurement
		5	50	250	500	1000	1500	2000	2500	3000	3500	4000	5500	6000	6500	
300	DC55M [△]	1.13	2.76	-	-	-	-	-	-	-	-	-	-	-	-	Very slight skin
	DC55M [△]	1.80	6.02	-	-	-	-	-	-	-	-	-	-	-	-	Very slight skin
	DC55M	-	-	-	15.32	16.96	-	-	-	20.53	-	-	-	-	-	Light skin formed
	DC55M	-	-	-	8.14	-	38.14	41.94	42.69	-	43.29	43.53	-	-	47.41	Light skin formed
	DC55M	-	-	-	20.01	23.13	24.43	-	25.06	25.74	-	-	28.19	-	-	Light skin formed
	WSX8788 [△]	0.67	1.47	-	-	-	-	-	-	-	-	-	-	-	-	Unchanged
	WSX8788 [△]	1.04	2.10	-	-	-	-	-	-	-	-	-	-	-	-	Unchanged
	WSX8788	-	-	-	4.12	6.43	-	-	-	12.78	-	-	-	-	-	Darkened
	WSX8788	-	-	-	11.34	16.01	19.48	-	20.06	21.70	22.04	-	-	29.41	-	Darkened
	SWS295	-	-	-	3.30	4.91	8.90	-	23.73	24.05	24.12	-	-	24.79	-	Light skin
	DC44	-	-	-	5.71	-	7.07	11.28	12.05	-	16.30	16.44	-	-	18.10*	High friction
	DC11	-	-	-	1.12	1.39	1.86	-	-	-	-	-	-	-	-	High friction
	SWS290	-	-	-	3.46	3.77	4.11	-	4.22	*	-	-	-	-	-	High friction
	B645	-	-	-	10.01	11.69	46.59	-	-	-	-	-	-	-	-	Very stiff
	A761	-	-	-	19.49	49.34	*	-	-	-	-	-	-	-	-	High friction
	MO40	-	-	-	18.15	21.61	*	-	-	-	-	-	-	-	-	Rock hard
	MO40	-	-	-	8.02	17.56	-	-	-	-	-	-	-	-	-	Rock hard
	SUPER-O	-	-	-	-	0.34	-	-	-	-	-	-	-	-	-	High friction
	SUPER-O	-	-	-	0.36	0.43	*	-	-	-	-	-	-	-	-	High friction
350	DC55M	-	-	-	22.13	28.27	-	-	-	39.72	-	-	-	-	-	Light skin
	DC55M	-	-	-	24.61	33.92	34.71	-	38.38	40.62	-	-	57.53	-	-	Firm skin
	WSX8788	-	-	-	13.59	23.76	-	-	-	47.61	-	-	-	-	-	Darkened
	WSX8788	-	-	-	12.39	24.33	26.74	-	43.26	54.78	-	-	75.63	-	-	Black, stiff
	SWS295	-	-	-	3.43	5.45	6.34	-	9.53	11.26	-	-	13.75	-	-	Light skin, high friction
	DC11	-	-	-	1.51	1.92	-	-	-	3.27	-	-	-	-	-	High friction
	SWS290	-	-	-	3.56	4.15	4.32	*	-	-	-	-	-	-	-	High friction
	B645	-	-	-	9.82	27.48	32.70	-	-	-	-	-	-	-	-	Very stiff
	A761	-	-	-	32.76	*	-	-	-	-	-	-	-	-	-	High friction
	MO40	-	-	-	18.08	-	-	-	-	-	-	-	-	-	-	Rock hard
	SUPER-O	-	-	-	0.05	0.65	0.69	-	-	-	-	-	-	-	-	High friction
	DC55M	-	-	-	32.70	40.24	-	-	-	85.90	-	-	-	-	-	Hard grey residue
400	DC55M	-	-	-	~30	*	-	-	-	-	-	-	-	-	-	Light skin darkened
	WSX8788	-	-	-	40.18	65.79	-	-	-	80.40	-	-	-	-	-	Hard brittle residue
	WSX8788 [△]	-	-	-	27.33	-	-	-	-	-	-	-	-	-	-	Darkened
	SWS295	-	-	-	16.02	-	-	-	-	-	-	-	-	-	-	Light skin
	DC11	-	-	-	2.05	2.82	-	-	-	10.45	-	-	-	-	-	Gummy
	SWS290	-	-	-	8.47	-	-	-	-	-	-	-	-	-	-	High friction
	A761	-	-	-	88.95	-	-	-	-	-	-	-	-	-	-	Hard
	MO40	-	-	-	31.35	*	-	-	-	-	-	-	-	-	-	Rock hard
	SUPER-O	-	-	-	△	-	-	-	-	-	-	-	-	-	-	Crystalized

Notes: [△], [△] Recently started tests to determine short-term losses and surface area effects, area ratio 3.2:1 ([△] [△])
[△] Weight loss not recorded, sample crystalized
[△] Used in friction test (Figure 64)
* Tests terminated.

c. Elastomer Compatibility Tests

A series of compatibility tests were conducted to determine the effects of selected lubricants on the test elastomers. Specimens of Buna N (No. N741-75, 366Y), silicone (No. S684-70), ethylene propylene (No. E692-75), and urethane (No. P648-90) elastomer O-rings were exposed to various greases at 150°F (Figure 67). At the end of 1000 hours, the elastomer specimens were removed, wiped free of grease, and their weights and hardnesses measured. Specimens exposed to DC55M and WSX8788 greases were continued to a total of 7000 hours at 150°F. Table 36 shows the results of these tests. Results after 1000 hours showed the DC55M grease softened the silicone and ethylene propylene elastomers, and caused noticeable increases in weight by dissolving in the elastomer. The SWS295 also softened the silicone elastomer, and caused a weight gain. This behavior was anticipated, since it is known that silicone greases, like DC55M and SWS295, dissolve in and soften the silicone elastomers. These tests were with elastomers immersed in grease, whereas only a light coat is applied for installation in components. The WSX8788 grease did not appear to affect any of the four elastomers significantly.

After 7000 hours, the silicone elastomers appeared to be nearly saturated with DC55M and still little affected by the WSX8788. The Minnesota Rubber nitrile, and to a lesser degree the Parker Seal nitrile, elastomers increased significantly in hardness for both DC55M and WSX8788 greases. The WSX8788 also caused significant weight gain in the Parker elastomer. The urethane elastomer soaked in WSX8788 broke, while the one soaked in DC55M cracked during handling for post-test measurements. It is not known whether the urethane failure and the increase in hardness of the nitriles was caused by the greases or was the result of long-term exposure at 150°F as no specimens were tested unlubricated.

Additional compatibility testing was conducted on two silicone elastomers in WSX8788, DC55M, and DC11 grease. Table 37 shows results after 572, 1200, and 4600 hours at 200 and 300°F. As expected, the silicone grease, DC11 and DC55M are less compatible and result in greater swelling of the silicone elastomers than the polyester-base WSX8788. Most of the reaction or swelling occurred during the initial 72 hours (results not shown).

Figure 67. Elastomer-Grease Compatibility Test
(1000 hr, 150°F)

TABLE 36
ELASTOMER - GREASE COMPATIBILITY TESTS

Grease	Total Time At Temperature (hr)	Temperature (°F)	Vendor†	Elastomer - Percent Change in Properties											
				Silicone			Nitrile (Buna-N)			EPR			Urethane		
				Weight	Hardness	Cross Section	Weight	Hardness	Cross Section	Weight	Hardness	Cross Section	Weight	Hardness	Cross Section
DC55M	7240	150	P	+28.76	-18.9	+11.4	+ 5.14	+24.7	+2.9	+6.00	+4.1	+2.4	-1.27	+4.2△	No change
			M				+ 1.36	+35.4	+0.5						
WSX8788	7240	150	P	+6.51	- 1.4	+3.3	+17.00	+21.9	+7.6	+2.13	+8.2	+1.0	-6.76	+4.3△	No change
			M				+ 9.38	+41.5	+2.3						
DC55M	1000	150	P	+23.17	-20.3	§	+ 3.43	+ 1.4	§	+9.94	-12.3	†	+0.007	+1.1	†
			M				+ 4.22	- 3.1	§						
WSX8788	1000	150	P	+2.63	- 1.4	§	+ 6.35	+ 2.7	§	+1.45	NC	†	-0.25	+1.1	†
			M				+ 5.58	+ 3.1	§						
SWS295	1000	150	P	+16.03	-13.5	§	- 0.48	+ 5.5	§	-2.41	+4.1	†	-1.19	+2.2	†
			M				- 5.09	+18.5	§						

Notes
 △ Elastomer cracked during handling for post test measurements
 △ Elastomer broke during handling for post test measurements
 Test discontinued
 † P = Parker, M = Minnesota Rubber
 § Value not recorded

"As Received" Elastomer Hardness (Shore A)
 Parker Seal Buna-N (N741-75) 72-75
 Parker Seal Silicone (S684-70) 73-74
 Parker Seal EPR (E692-75) 71-74
 Parker Seal Urethane (P648-50) 91-94
 Minnesota Rubber Nitrile (366Y) 64-66

TABLE 37
LUBRICANT EFFECT ON SILICONE SEALS

Temperature (°F)	Grease	Vendor*	Percent Change As A Function of Time								
			572 Hours [†]			1200 Hours [†]			4646 Hours [†]		
			Weight	Hardness	Cross Section	Weight	Hardness	Cross Section	Weight	Hardness	Cross Section
200	DRY	P	- 0.2	NC	-3.3	<1.0	+ 1.0	-3.3	- 0.20	+ 4.2	- 2.8
		M	- 0.8	NC	-0.4	<1.0	- 3.0	-2.4	- 0.96	+ 7.0	- 1.4
	DC55M	P	+23.6	-14.0	+7.1	+26.6	-15.6	+6.2	+29.94	-14.3	+10.4
		M	+17.9	- 7.0	+5.7	+16.1	-21.0	+3.8	+20.02	-21.1	+ 8.0
	DC11	P	+ 7.9	- 7.0	+1.4	+ 9.8	- 9.5	+2.4	+12.77	- 9.9	+ 4.8
		M	+ 7.3	- 4.0	+2.4	+ 8.7	- 7.0	+2.3	+12.03	-12.7	+ 5.3
	WSX8788	P	+ 3.9	- 1.0	+1.9	+ 3.9	+ 3.0	-1.4	+ 7.42	+ 4.3	NC
		M	+ 1.5	- 7.0	-1.4	+ 0.7	+ 7.0	-2.3	- 0.36	+ 7.0	- 0.9
300	DRY	P	- 0.5	NC	-2.4	- 0.6	+ 1.4	-1.4	- 0.73	+11.1	- 1.9
		M	- 1.5	+ 7.0	-0.4	- 1.3	+ 6.0	-1.4	- 1.77	+11.3	- 1.9
	DC55M	P	+26.8	-20.0	+7.0	+28.6	-21.4	+5.7	+40.08	-28.6	+12.3
		M	+18.2	-21.0	+3.2	+18.8	-20.7	+2.8	+23.38	-28.2	+ 8.4
	DC11	P	+ 7.8	- 7.0	+1.9	+10.0	- 9.7	+1.9	+16.22	-11.3	+ 4.8
		M	+ 5.1	- 3.0	+1.4	+ 6.5	+ 1.4	-1.4	+ 9.80	-14.1	+ 3.8
	WSX8788	P	+ 5.8	<1.0	+1.4	+ 5.2	+ 2.9	-1.0	+ 8.09	+ 1.4	+ 1.1
		M	+ 0.1	NC	NC	- 0.6	NC	-1.4	- 2.79	- 1.4	- 2.3

Notes: *P = Parker; M = Minnesota Rubber
†DC11 tested 72 hr less

"As Received" Elastomer Hardness
Parker Seal (S684-70) 73-74 (Shore A)
Minnesota Rubber (71417) 70-71

5. Elastomer Aging and Shelf Life*

a. Aging

Deterioration with time or aging relates to the nature of the elastomer molecule, a long chain-like structure consisting of many smaller molecules joined together. At least three principle types of reactions are associated with aging. They usually occur concurrently, but in varying degrees.

- 1) Scission — The molecular bonds are cut, dividing the chain into smaller segments. Ozone, ultraviolet light, and radiation cause degradation of this type.
- 2) Cross Linking — An oxidation process, whereby additional inter-molecular bonds are formed. This process may be a regenerative one. Heat and oxygen are principle causes of this type of attack.
- 3) Modification of Side Groups — A change in the complex, weaker fringe areas of the molecular construction due to chemical reaction. Moisture, for example, could promote this activity.

Note that all mechanisms by which rubber deteriorates with time are attributable to environmental conditions. It is environment then, not age, that is significant to seal life, both in storage and actual service.

Normal procedure throughout the seal industry is to indicate the date of cure on the seal package by quarter and calendar year (e. g., 1 Q 73 would indicate the first quarter of 1973). Thus, the elastomer seal user is informed of the age of the seals at the time of receipt.

b. Shelf Life

Shelf life varies with the resistance of each synthetic elastomer to normal storage conditions. Military Handbook 695 (MIL-HDBK-695) sorts synthetic elastomers into three groups, according to "age resistance generally associated with products fabricated from various rubbers." These groups consist of materials suitable for periods of storage as noted in Table 38.

Where the non-age-sensitive elastomers, mentioned in Table 38, in 10- or 20-year categories, are involved, considerable storage life without detectable

*Parker Seal Co. "Seal Compound Manual," (December 1971)

TABLE 38
SHELF LIFE OF ELASTOMERIC MATERIALS

Type Rubber	Common or Trade Name
	Maximum (Up to 20 years)
Fluorosilicone	Silastic LS
Polyacrylate	Acrylate
Polysulfide	Thikol
Silicone	Silastic, Silicone
	Average (5 to 10 years)
Chlorosulfonated polyethylene	Hypalon
Ethylene-propylene-diene	Ethylene propylene terpolymer
Fluorocarbon	Fluorel, Viton
Isobutylene-isoprene	Butyl, GR-1
Polychloroprene	Neoprene, GR-M, Chloro- prene
Polyvinyl chloride	Vinyl, Poly (vinyl chloride)
Polyurethane	Urethane
	Minimum (2 to 5 years)
Butadiene-acrylonitrile	Nitrile, Buna N
Butadiene-styrene	CR-S, Buna S
Cis polybutadiene	Stereo polybutadiene
Natural	Pale crepe, smoked sheets

damage is common, even under adverse conditions. For materials mentioned in the 2- to 5-year category, which are subject to deterioration, the following conditions are suggested for maximum life:

- 1) Ambient temperature not exceeding 120°F (49°C)
- 2) Exclusion of air (oxygen)
- 3) Exclusion of contamination
- 4) Exclusion of light (particularly sunlight)
- 5) Exclusion of ozone generating electrical devices
- 6) Exclusion of radiation.

Generally, polyethylene bags stored in larger cardboard containers or polyethylene-lined craft paper bags ensure optimal storage life. However, in normal warehousing conditions, life of even the relatively age-sensitive elastomers is considerable.

In order to minimize the deterioration of elastomer seals used in the Seal Test Program, all seals are currently stored in airtight, friction-top steel containers . Whenever quantities of seals are procured and immediate use is not planned, it is recommended that adequate storage be provided.

B. STATIC TESTING

Fabrication of three Static O-ring Demonstration Test fixtures and three Static Information Test fixtures was completed in the AI shop. Each Demonstration Test fixture (Figure 68) tests one pair of O-ring seals at the FFTF design conditions (50% compression and 16 rms finish). Each Information Test fixture tests four pairs of seals at off-design conditions (10 and 20% compression, 8 and 32 rms finish, and with and without lubricant). Figure 69 shows the test fixtures and valve-gage panel.

Designs of the Static Inflatable Seal Test and Static Molded-In-Metal Seal Test fixtures were completed and the drawings were reviewed, approved, and released to storage until ARD approval to proceed is obtained.

1. Demonstration Tests

Three static demonstration tests (No. 1, 2, and 3) were assembled with pairs of Buna N, EPR, and silicone elastomer O-ring seals , as shown in Table 39, and static leakage testing was begun at 150, 150, and 300 °F, respectively. The preliminary leakage data for the Buna N seals appear to be of the same order of magnitude as permeation type, calculated for a seal under 50% compression, but the leakage values for the EPR and silicone seals appear to be low. These low values were attributed to the high losses through the outer, containment seal. These losses were not accounted for in the initial method of calculation, but, as stated earlier, the computer program was later revised to correct for these losses.

After these tests had run for three months, Tests 2 (EPR) and 3 (Silicone)^{*} were shut down to replace the outer containment seals with Buna N seals of lower permeability.

^{*}Note: Although Buna N is not normally used at 300 °F, it was decided that in this static application, it would provide a satisfactory "one-time" seal.

9006-40116

Figure 69. Static Seal Test Fixtures

TABLE 39
STATIC DEMONSTRATION TESTS
(O rings only)

Test	Seals	Compressions (%)	Gland Finish (rms)	Temper- ature (°F)	Lubricant	Comments
1	Buna N (N741-75) Parker Seal Co.	50	16	150	DC55M	Test completed. No observed leakage
2	EPR (E692) Parker Seal Co.	50	16	150	DC55M	Test completed. No observed leakage
3 Rerun	Silicone (S684) Parker Seal Co.	50	16	300	DC55M WSX8788	Terminated after 3 months Test in progress for 6 months
4	Urethane (P642) Parker Seal Co.	50	16	100	DC55M	Tests in progress
5	Urethane (522FX) Minnesota Rubber	50	16	100	DC55M	Tests in progress

When Static Demonstration Test 3 was disassembled to remove the Silicone O-rings, it was found that the plates were tightly stuck together, and a wedge had to be used to force the plates apart. The silicone O-rings were firmly attached to the stainless-steel plates, causing small pieces of seal to tear off as the plates were separated. The seal was stuck more firmly to the top plate than to the seal gland. As the seal was pulled from the top plate, the seal tore partially in several places, and, at one place, tore completely across the cross section. A post-test photograph of the seals is shown in Figure 70. Pieces of seal, stuck to and remaining in the seal glands, are evident in the photo of the fixture, Figure 71.

The fixture plates were cleaned, and new seals installed (two Parker silicone test O-rings, one Minnesota Rubber Buna N containment O-ring). It was decided not to use the DC55M lubricant on these seals, but to use the WSX8788 lubricant. The test was reassembled, and the leakage measurements restarted. The test will be completed in mid-July after 6 months of test.

In April 1974, the seal program was redirected to increase the extent of urethane testing, since no static urethane tests were planned in the initial program. Static Tests 4 and 5 were set up with urethane O-rings from Parker Seal and Minnesota Rubber. These seals are currently in test at 100°F under 50% compression.

2. Information Tests

Static information tests were set up with Parker Seal Company and Minnesota Rubber Company Buna N, EPR, and silicone elastomer rings under 10 and 20% compression, as noted in Table 40 and illustrated in Figure 72. Static Information Tests 2A-D and 3A-D showed lower than anticipated leakage values, as had the corresponding demonstration tests on the same materials. Test 3 was shut down, and the outer containment removed and replaced with a Buna N seal. (Test 2 was not interrupted for a seal replacement, as discussed in the next section.) When Static Information Test 3A-D was disassembled to replace the outer seals, the silicone O-rings were found to be adhering to the steel plates in the same manner as when Static Demonstration Test 3 was disassembled. The inner test silicone seals were not found to be stuck to the plates, probably

9006-40155

Figure 70. Silicone Test Seals, Static
Demonstration Test 3 (Parker Sili-
cone O Ring, Dow Corning 55M,
3 months, 300°F)

9006-40153

Figure 71. Seal Particles Torn from Test Seals,
Static Demonstration Test

TABLE 40
STATIC INFORMATION TESTS
(O rings only)

Test	Seals	Compressions (%)	Gland Finish (rms)	Temperature (°F)	Lubricant	Comments
Information 1A	Buna N (366Y) Minnesota Rubber Co.	10	8	150	DC55M	Test completed no leakage observed.
Information 1B	Buna N (N741-75) Parker Seal Co.	10	32	150	DC55M	Test completed no leakage observed.
Information 1C	Buna N (N741-75) Parker Seal Co.	20	8	150	DC55M	Test completed no leakage observed.
Information 1D	Buna N (366Y) Minnesota Rubber Co.	20	32	150	DC55M	Test completed no leakage observed.
Information 2A	EPR (E692) Parker Seal Co.	10	8	150	DC55M	Test completed no leakage observed.
Information 2B	EPR (559N) Minnesota Rubber Co.	10	32	150	DC55M	Test completed no leakage observed.
Information 2C	EPR (559N) Minnesota Rubber Co.	20	8	150	DC55M	Test completed no leakage observed.
Information 2D	EPR (E692) Parker Seal Co.	20	32	150	DC55M	Test completed no leakage observed.
Information 3A	Silicone (71417) Minnesota Rubber Co.	10	8	300	DC55M	Test completed no leakage observed.
Information 3B	Silicone (S684) Parker Seal Co.	10	32	300	DC55M	Test completed no leakage observed.
Information 3C	Silicone (S684) Parker Seal Co.	20	8	300	DC55M	Test completed no leakage observed.
Information 3D	Silicone (71417) Minnesota Rubber Co.	20	32	300	DC55M	Test completed no leakage observed.

6507-40474

Figure 72. Static Information Seal, Test Fixture Layout

because these seals were compressed only 10 and 20%. All seals were removed, measured, etc. The test was restarted and run for an additional 3 months. Nothing unusual was noted when Static Information Tests 1A-D and 2A-D were disassembled; however, when Test 3A-D was disassembled the Parker Seal Buna N (N741) test fixture containment O-rings were found to be split in several places (Figure 73). The Minnesota Rubber Buna N (366Y) fixture O-rings, also subjected to 300 °F under the same 45% compression, were found to be almost completely flat (~100% compression set), as indicated in Figure 74.

It is interesting to note that, even though one seal was split in several places and the other seal had suffered excessive set, no seal leaked test gases to the atmosphere at 300 °F and 0.5-psig pressure, nor did the seals leak when the fixture was cooled to room temperature.

3. Leakage Calculations

Leakage values can be calculated knowing the seal geometry, % compression, temperature, and material permeability. Table 41 compares these calculated values with the measured values for both H₂ and Kr (Xe is not used in the static tests). Generally, only good agreement is obtained with the Buna N seal materials. It had been recognized early in the test program that the loss of gas by permeation through the outer, containment O-ring seal would produce low leakage values for the higher permeability materials, but that this loss could be corrected to produce a more accurate leakage value.

The computer program was modified to calculate the seal leakage value on the basis of the observed readings. Using this approximate value, the amount of gas leaking through the outer containment seal is calculated. The amount of gas lost through the outer seal is added to the amount of gas measured in the collector chamber, and a higher leakage value calculated. The iteration process continues for several steps until the amount of change is insignificant, and the final leakage value is calculated. Also incorporated in the program is a material balance sequence, and the necessary temperature and pressure corrections to reduce the leakage values to std cc of gas/sec/linear in. of seal/atm ΔP .

Using the new program, the measured leakage values were calculated for the seal tests. The values for the STA D1 and STA D2 (50% compression)

Figure 73. Static Information Test 3 Rerun, 300°F,
Region C

9006-40210

Figure 74. Static Information Test 3 Rerun, 300°F,
Region A

9006-40209

TABLE 41
STATIC O-RING SEAL TESTS - LH₂ AND L_{Kr} VALUES
FROM ELASTOMER SEAL LEAKAGE TESTS

Seal Type	Elastomer	Supplier	Test Type	Compression (%)	Finish (rms)	Lubricant	Leakage*			
							L _{H₂}		L _{Kr}	
							Meas.	Calc.	Meas.	Calc.
O	Buna N [†]	PS	STA-D-1	50	16	DC55M	32	23	5	6
O	Buna N	MR	STA-I-1A	10	8	DC55M	82	114	32	33
O	Buna N	PS	STA-I-1B	10	32	DC55M	61	78	4	20
O	Buna N	PS	STA-I-1C	20	8	DC55M	49	62	46	16
O	Buna N	MR	STA-I-1D	20	32	DC55M	40	90	13	26
O	EPR [†]	PS	STA-D-2	50	16	DC55M	90	90	35	38
O	EPR	PS	STA-I-2A	10	8	DC55M	120	315	70	130
O	EPR	MR	STA-I-2B	10	32	DC55M	135	420	60	170
O	EPR	MR	STA-I-2C	20	8	DC55M	210	320	75	130
O	EPR	PS	STA-I-2D	20	32	DC55M	110	230	60	96
O	Silicone [§]	PS	STA-D-3	50	16	WSX 8788	470	3,265	104	1,700
O	Silicone	MR	STA-I-3A	10	8	DC55M	**	19,680	**	8,000
O	Silicone	PS	STA-I-3B	10	32	DC55M	**	11,520	**	6,000
O	Silicone	PS	STA-I-3C	20	8	DC55M	**	8,640	**	4,500
O	Silicone	MR	STA-I-3D	20	32	DC55M	**	14,760	**	6,000

EPR = Ethylene propylene rubber

PS = Parker Seal

MR = Minnesota Rubber

L_{Kr} = Std cc/sec/linear in. of seal/atm ΔP of Kr x 10⁻⁸

LH₂ = Std cc/sec/linear in. of seal/atm ΔP of H₂ x 10⁻⁸

*Listed values corrected for permeation leakage through outer fixture seal

† = 150°F § = 300°F **To be calculated

showed very good agreement, but the values for the 10 and 20% compression tests were lower than anticipated if the leakages were calculated from permeation and geometry factors.

The same calculation method, used on the silicone seals, of the STA D-3 test gave a value much lower than that anticipated from permeation type calculations, that is, the measured values continue to be about 1/10 of the values calculated from $\bar{P}$ data. The silicone seal information test data have not been recalculated with the new program at this time.

4. Special Silicone Seal Test

Because of the inability to approximate the leakage values for silicone O-rings calculated from permeation data, a special test was set up to measure the permeation of Kr gas through silicone rubber seals at room temperature ($\sim 75^\circ\text{F}$). A static seal test fixture was used with a modified gas piping, as shown in Figure 75. The Ar/Kr gas in the outer cavity ① was used to minimize the loss of Ar/Kr gas from the middle cavity ② to the outer cavity, restricting the permeation of the Kr essentially to the Ar-filled center cavity ③. The total gas pressure in each cavity was the same at the start of the test, although the pressure in the outer cavity decreased during the test and was periodically replenished.

The test was conducted for 215 hr. The measured L_{Kr} for the silicone O-ring (Parker Seal Compound S684) was found to be 230×10^{-8} std cc/sec/linear in./atm ΔP compared to a value calculated from $\bar{P}$ values of 600×10^{-8} . This test not only substantiates the low values obtained in the static tests, but points the need for further special tests on silicone rubber.

A review of the two tests — the basic permeation test on a slab specimen and the functional seal leakage test of an O-ring — implies that the parameter of time may be the cause of the large apparent difference in permeation leakage in the silicone rubber. That is, the permeation test is conducted for only several hours, whereas the leakage test continues for several hundreds of hours. It is assumed in the permeation test that the constant rate (expressed in cc/sec) of gas permeation, attained during several hours of testing, will remain constant indefinitely if the driving force is constant and the collection reservoir is sufficiently large. Further testing is needed to establish conclusively the time characteristics of permeation.

Figure 75. Special Static Seal Test

5. Compression Set

Compression set testing was conducted on numerous elastomer O-ring compositions from Parker Seal Co. and Minnesota Rubber Co. to gain an insight into the sealing ability of the O-rings after long periods of time under compression at various temperatures.

O-rings, 3/16-in. cross section by 4 in. OD were installed in grooved flanges, fabricated per AI Drawing N707800034, which apply a 50% compression. The test fixtures were placed in air atmosphere ovens and heated at temperatures of 100 to 300 °F, depending upon the elastomer tested. The fixtures were removed after 1, 2, 4, 8, 16, and 32 weeks, cooled to room temperature, and opened. The seal dimensions were measured for comparison to the starting values. Figures 76 through 85 show the compression set values obtained on 10 elastomer compounds.

Compression set testing of the Parker Seal Buna N (N741-75) O-rings was interrupted when it was found that the O-rings were cracked after 1 week of testing at 150 and 200 °F under 50% compression. Figure 86-a shows the two seals, and Figure 86-b shows a close-up of the crack in the 150 °F seal. The test was repeated with two new seals, which cracked after 2 weeks of testing. The vendor was contacted on the problem, and the tentative conclusion was that the compression was too high for the high-modulus and high-hardness rubber (Shore A 75). (Note: About 50% compression is used in selected IVHM applications to minimize seal leakage-permeation.)

Earlier, this elastomer (Buna N N741-75) had been recommended to replace butyl rubber seals in the FFTF, because of the poorer radiation resistance of the butyl rubbers. On the basis of this latest information, however, it was recommended that the alternate composition, Minnesota Rubber Buna N (366Y), be used in place of the Parker Buna N, at least for high seal compression applications. The Minnesota Rubber Buna N is slightly softer, but has completed 32 weeks testing at 100, 150, and 200 °F without evidence of damage.

The 80 and 95% confidence limits have been calculated for several of the completed tests. Tables 42 through 47 list these calculated values, and Figures 87 and 88 graphically show these limits for a urethane and Buna N elastomer. As tests are completed, the corresponding limits will be calculated for the test data.

6507-40477

Figure 76. Compression Set of Minnesota Rubber O-Ring Seals, Buna N, Compound 366 Y

Figure 77. Compression Set of Parker Seal O-Ring Seals, Buna N, Compound N741

9006-4096

Figure 78. Compression Set of Minnesota Rubber O-Ring Seals, Urethane, Compound 522 FX

6507-40479

Figure 79. Compression Set of Parker Seal O-Ring Seals, Urethane, Compound P642

6507-40480

Figure 80. Compression Set of Parker Seal O-Ring Seals, Urethane, Compound P648

6507-40481

Figure 81. Compression Set of Parker Seal O-Ring Seals, EPR, Compound E529

6507-40482

Figure 82. Compression Set of Minnesota Rubber O-Ring Seals, EPR, Compound 559N

9006-4097

Figure 83. Compression Set of Parker Seal O-Ring Seals,
Ethylene Propylene, Compound E692

Figure 84. Compression Set of Minnesota Rubber O-Ring Seals,
Silicone, Compound 71417

9006-4098

Figure 85. Compression Set of Parker Seal O-Ring Seals, Silicone, Compound S684

9006-40114A

a. Seals Tested at 150 and 200°F

9006-40121A

b. Close-up of Crack in 150°F Seal

Figure 86. Parker Seal Buna N (N741-75) O-Ring Seals
after 1 Week under 50% Compression

TABLE 42

CONFIDENCE LIMITS - TEST DATA FOR COMPRESSION SET OF PARKER
SEAL EPR O-RINGS (E692)

Temperature (°F)	Time (hr)	Compression Set		Confidence Limits			
		Measured	Calculated	80%		95%	
100	192	17.1	17.1	15.1	19.5	13.6	21.6
	324	20.9	19.3	17.2	21.8	15.6	24.0
	720	21	23.2	20.8	26.0	19.0	28.5
	1344	26.3	26.9	24.0	30.1	21.9	33.0
	2688	32.7	31.5	28.0	35.5	25.5	39.1
	5376	37.5	37.0	32.6	42.1	29.3	46.7
150	192	31.6	31.4	27.9	35.4	25.4	38.9
	324	38.2	37.4	33.5	41.8	30.6	45.7
	720	45.3	48.8	44.0	54.2	40.4	59.0
	1344	60.8	60.1	54.2	66.7	49.8	72.7
	2688	82.5	75.8	67.9	84.5	62.1	92.4
	5376	91	95.4	84.7	107.5	76.9	118.4
200	To be calculated						

TABLE 43

CONFIDENCE LIMITS - TEST DATA FOR COMPRESSION SET OF
MINNESOTA RUBBER BUNA N O-RINGS (366Y)

Temperature (°F)	Time (hr)	Compression Set		Confidence Limits			
		Measured	Calculated	80%		95%	
100		In Test					
150	168	15	15.5	13.4	18.0	11.9	20.2
	336	22.2	21.3	18.7	24.4	16.7	27.2
	1008	33.3	35.4	31.2	40.1	28.2	44.4
	2016	54.8	48.6	42.8	55.2	38.6	61.2
	4200	66.7	68.0	59.5	77.9	53.4	86.9
	5376	73	76.3	66.4	87.6	59.4	97.9
200	To be calculated						

TABLE 44
CONFIDENCE LIMITS - TEST DATA FOR COMPRESSION SET OF PARKER
SEAL EPR O-RINGS (E6 92)

Temp (°F)	Time (hr)	Compression Set		Confidence Limits			
		Measured	Calculated*	80%		95%	
150	168	6.1	8.2	5.3	12.7	3.7	18.2
	336	11.7	9.7	6.5	14.5	4.7	20.1
	672	13.2	11.4	7.8	16.8	5.7	22.9
	1344	15.5	13.5	9.2	19.8	6.7	27.0
	2688	16.5	15.8	10.6	23.7	7.6	32.8
	5376	15.1	18.7	12.1	28.9	8.5	41.1
200	168	17.7	17.4	15.1	20.1	13.4	22.6
	336	21.9	22.4	19.6	25.6	17.6	28.5
	672	29.9	28.8	25.4	32.7	22.9	36.3
	1344	33.6	37.1	32.6	42.1	29.4	46.7
	2688	52.8	47.7	41.7	54.5	37.4	60.7
	5376	59.3	61.3	53.0	70.8	47.1	79.6
250	168	26.4	27.0	24.3	30.0	22.2	32.7
	336	34.1	34.2	31.0	37.7	28.6	40.8
	672	43.7	43.3	39.4	47.5	36.5	51.3
	1344	55.2	54.8	49.9	60.2	46.3	64.9
	2688	75.3	69.4	62.9	76.5	58.1	82.9
	5376	81.7	87.9	79.1	97.7	72.5	106.5

*Least squares fit.

TABLE 45
CONFIDENCE LIMITS - TEST DATA FOR COMPRESSION SET OF PARKER
SEAL SILICONE O-RINGS (S684)

Temp (°F)	Time (hr)	Compression Set		Confidence Limits			
		Measured	Calculated ¹⁾	80%		95%	
200	168	16.2	15.6	13.5	17.9	12.1	20.0
	336	21.1	20.4	18.0	23.2	16.2	25.8
	672	25.6	26.9	23.7	30.4	21.5	33.5
	1344	31.5	35.3	31.2	39.9	28.2	44.1
	2688	48.2	46.3	40.8	52.7	36.7	58.5
	5376	64.0	60.9	53.0	70.0	47.3	78.4
150	168	8.2	10.74	6.67	17.30	4.53	25.47
	336	13.6	11.84	7.63	18.37	5.34	26.24
	672	16.5	13.05	8.58	19.85	6.10	27.90
	1344	13.6	14.38	9.45	21.88	6.72	30.75
	2688	19.4	15.85	10.21	24.59	7.15	35.12
	5376	13.6	17.46	10.84	28.13	7.37	41.41
250	168	27.4	26.65	21.32	33.31	17.79	39.92
	336	33	35.45	28.85	43.54	24.42	51.45
	672	44.2	47.15	38.74	57.39	33.04	67.30
	1344	69.5	62.72	51.53	76.34	43.94	89.52
	2688	95.5	83.43	67.92	102.49	57.48	121.10
	5376	97.5	110.98	88.78	138.73	74.09	166.25

*Least squares fit.

TABLE 46
CONFIDENCE LIMITS – TEST DATA FOR COMPRESSION SET OF
PARKER SEAL EPR O-RINGS (E259)

Temp (°F)	Time (hr)	Compression Set		Confidence Limits			
		Measured	Calculated*	80%		95%	
150	To be tested						
200	168	5.8	In test				
	336	8.6					
250	168	54.3	54.0	53.2	55.0	52.3	55.8
	336	62.3	62.6	61.7	63.6	60.8	64.5
	672	72.0	72.5	71.5	73.6	70.5	74.6
	1344	84.8	84.0	82.7	85.3	81.5	86.4
	2688	97.0	97.3	97.3	95.6	98.9	100.5

*Least squares fit.

TABLE 47
CONFIDENCE LIMITS – TEST DATA FOR COMPRESSION SET OF
MINNESOTA RUBBER SILICONE (71417) O-RINGS

Temp (°F)	Time (hr)	Compression		Confidence Limits			
		Measured	Calculated*	80%		95%	
150	To be tested						
200	168	12.9	To be calculated				
	336	19.1					
	672	22.0					
	1344	23.4					
	2856	38.3					
	5376	40.6					
250	168	26.5	26.9	24.2	29.9	22.3	32.5
	336	32.3	33.5	30.5	36.9	28.2	39.9
	672	45.2	41.8	38.1	45.8	35.4	49.3
	1344	50.7	52.1	47.5	57.1	44.1	61.5
	2688	67.7	64.8	58.9	71.4	54.5	77.2
	5376	77.5	80.8	72.8	89.7	66.9	97.6

*Least squares fit.

6507-40483

Figure 87. Compression Set of Minnesota Rubber O-Ring Seals, Urethane, Compound 522 FX, with Confidence Limits

6507-40484

Figure 88. Compression Set of Minnesota Rubber O-Ring Seals, Buna N, Compound 366Y, with Confidence Limits

6. Useful Life Estimate

Compression set data were plotted on an Arrhenius-type graph of log time (weeks) vs $1000/T^{\circ}\text{K}$, in an effort to more accurately predict the maximum temperature for a 5-year useful life. Figures 89 through 92 show the graphical results. Urethane (522FX), Buna N (N-219), EPR (E692), and silicone (S684) exhibit maximum-use temperatures (5-year life) of 120, 106, 189, and 189°F, respectively, at a 90% compression set limit. Lowering the compression set limit to 80% decreases the maximum-use temperature by 15°F or less. It is interesting to note that, in each figure, the data points at the high-temperature test conditions depart from the straight line, indicating that some physical change is occurring in the elastomer, causing a deviation from the linear.

Estimation of the Minnesota Rubber Buna N (366Y) useful life cannot be predicted until additional data are obtained, but it appears from an extrapolation of the compression set data (Figure 76) that a 5-year life will be limited to less than 100°F.

7. Other Compression Set Data

The 300°F compression set values for the 12 silicone seals removed from Static Information Tests 3A through 3D were determined from the seal dimensions. Table 48 shows the compression set values, and the pre- and post-test hardness values. It can be seen that the Parker Seal silicone O-rings exhibited significantly greater set than the Minnesota Rubber silicone O-rings. These data confirm the observations at 200 and 250°F, which also showed the Minnesota Rubber Silicone compound to exhibit less set. It also is seen that the seals compressed at only 10 and 20% exhibited much less set than those seals compressed 45%. Earlier test data had shown that no difference in % set could be seen between seals compressed 35 and 50%. It is possible that the amount of set is roughly proportioned to the % compression at lower compression, but is not proportional to the % compression after a certain level is reached.

Even though the observed set was ~100%, no leakage was observed across the test seal with 0.5-psi argon differential. The fact that the seal was well adhered to the steel would contribute to the nonleak observation.

9006-40134A

Figure 89. Estimation of Seal Useful Life at 80 and 90% Compression Set – Minnesota Rubber O-Ring Seal, Urethane, Compound 522 FX

9006-40135

Figure 90. Estimation of Seal Useful Life at 80 and 90% Compression Set – Parker O-Ring Seal, Buna N, Compound N219

Figure 91. Estimation of Seal Useful Life at 80 and 90% Compression Set – Parker O-Ring Seal Ethylene Propylene Compound E692

9006-40137

Figure 92. Estimation of Seal Useful Life at 80 and 90% Compression Set - Parker O-Ring Seal, Silicone Compound S684

TABLE 48
COMPRESSION SET AND HARDNESS OF SILICONE
O-RINGS FROM STATIC INFORMATION TEST 3
(1386 hr - 300°F)

Vendor	Seal Diameter (in.)	Compression (%)	Compression Set (%)	Hardness	
				Pre	Post
Minnesota Rubber Compound 71417	2	10	36.4	72	65
	4	10	31.6	71	65
	8	45	91.5	72	59
	2	20	20.9	65	59
	4	20	37.3	70	54
	8	45	98	75	52
Parker Seal Compound S684	2	10	61.5	75	66
	4	10	68.5	75	68
	8	45	100	75	70
	2	20	74.4	76	65
	4	20	83.8	74	65
	8	45	99	74	69

TABLE 49
COMPRESSION SET AND HARDNESS OF EPR O-RINGS
FROM STATIC INFORMATION TEST 2
(2154 hr, 150°F)

Vendor	Seal Diameter (in.)	Compression (%)	Compression Set (%)	Hardness	
				Pre	Post
Parker Seal Compound E692	2	10	11.8	74	75
	4	10	33.3	76	75
	8	45	18.3	65	61
	2	20	16.7	76	76
	4	20	17.8	74	74
	8	45	20.2	65	63
Minnesota Rubber Compound 559N	2	10	20.0	68	69
	4	10	31.8	73	72
	8	45	15.8	70	70
	2	20	28.0	68	69
	4	20	20.0	71	73
	8	45	11.3	68	70

The 150°F compression set values for the 12 EPR seals removed from Static Information Tests 2A through 2D are shown in Table 49 along with the set values and the pre- and post-test hardnesses. No significant difference was observed, in this case, between the % set at 10 and 20% compression and the % set at 45% compression. This is different from the data on silicone, where less set accompanied less compression.

The 300°F compression set values for the Buna N and silicone O-rings from Static Information Test 3 (Rerun) are listed in Table 50. The silicone seals showed compression sets ranging from 42 to 67% after the 3 months exposure at 300°F under 10 and 20% compression. The Minnesota Rubber and Parker Seal Buna N O-rings had about 95 and 60% set, respectively; but as noted earlier in Figure 73, the Parker seal was split in several places. This test confirmed the earlier conclusion that high hardness, high module seals should not be used at high compression (Section III-B-5).

C. PERMEABILITY TESTS

Permeability tests were continued on slab samples of elastomers of the same compounds from which the test seals were fabricated. The permeability data were treated with a computerized "least squares" calculational method to obtain the "best fit". These refined plots of log permeability is $1/T^{\circ}K$ for elastomers are shown in Figures 93 through 114. Generally, the Greene Tweed and the Parker Seal Company compounds have slightly lower permeabilities than the Minnesota Rubber compounds, although in many cases the differences are probably less than the probable error of about $\pm 20\%$. Figure 112 through 114 are repeats of permeabilities conducted earlier (Figure 94, 95, and 103, respectively) on slab samples of Buna N and silicone elastomer from different lots and batches to establish repeatability. Tables 51 and 52 compare the "smoothed" values for the least square fit relationship. In general, the variation between samples is not large, except in the H_2 values which are not too repeatable. It is not unlikely that variations in the H_2 values are real, and not experimental error. Elastomers are generally unsaturated materials which possess large numbers of double bonds ($-C=C-$ groups), and it is feasible that the migration of the H_2 is influenced by these sites by the reaction of the double bond with the H_2 .

TABLE 50
 COMPRESSIONS SET OF SEALS REMOVED FROM STATIC INFORMATION
 TEST NO. 3 (RERUN) AFTER 2184 HOURS

Elastomer	Diam (in.)	Location	Com- pression* (%)	Vendor	Hardness		Com- pression Set (%)
					Pre	Post	
Buna N	8	A	44.3	MR	70	82	96.8
Buna N	8	B	44.7	PS	75	84	59.6
Buna N	8	C	44.7	PS	76	85	56.7
Buna N	8	D	45.2	MR	71	80	94.7
Silicone	4	A	9.0	MR	74	74	65.5
Silicone	4	B	8.6	PS	77	74	50.0
Silicone	4	C	19.5	PS	76	74	62.2
Silicone	4	D	20.0	MR	75	72	66.7
Silicone	2	A	9.0	MR	72	70	42.1
Silicone	2	B	8.6	PS	76	75	46.7
Silicone	2	C	19.5	PS	75	74	61.1
Silicone	2	D	20.4	MR	73	67	47.7

*For a nominal 0.210 in. cross section O-ring

9006 40100

Figure 93. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Buna-N Rubber, Greene-Tweed Co., Compound 969

9006-40101

Figure 94. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Minnesota Rubber Co., Buna N, Compound 366Y

9006-40102

Figure 95. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Buna N Rubber, Parker Seal Co., Compound N741

9006 40103

Figure 96. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Urethane Rubber, Greene Tweed Co., Compound 379

Figure 97. $\log \bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Urethane Rubber, Minnesota Rubber Co., Compound FX522

Figure 98. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Urethane Rubber, Parker Seal Co., Compound P648

9006 40106

Figure 99. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Ethylene-Propylene Rubber, Greene-Tweed Co., Compound 972

9006-40107

Figure 100. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Ethylene Propylene Rubber, Minnesota Rubber Co., Compound 559N

9006-40108

Figure 101. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Ethylene Propylene Rubber, Parker Seal Co., Compound E692

Figure 102. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Silicone Rubber, Greene-Tweed Co., Compound 407

Figure 103. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Silicone Rubber, Minnesota Rubber Co., Compound 71417

9006-40111

Figure 104. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Silicone Rubber, Parker Seal Co., Compound S684

6507-40469

Figure 105. Log $\bar{P}$ vs 1/T (°K) for H₂, Ar, Kr, and Xe for Butyl Rubber, Parker Seal Co., Compound B318-70

9006-40144

Figure 106. Log $\bar{P}$ vs $1/T$ (°K) for H₂, Ar, Kr, and Xe for Urethane Parker Seal Co., Compound P642

Figure 107. Log $\bar{P}$ vs 1/T (°K) for H₂, Ar, Kr, and Xe for EPR, Parker Seal Co., Compound E529

Figure 108. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Buna S Minnesota Rubber Co., Compound 480DR

9006-40143

Figure 109. Log $\bar{P}$ vs $1/T$ (°K) for H₂, Ar, Kr, and Xe for Buna S, Parker Seal Co., Compound G244

9006-40141

Figure 110. Log $\bar{P}$ vs $1/T$ (°K) for H₂, Ar, Kr, and Xe for Polyacrylic, Minnesota Rubber, Compound 335GA

Figure 111. Log $\bar{P}$ vs $1/T$ (°K) for H₂, Ar, Kr, and Xe for Polyacrylic, Parker Seal Co., Compound A607

6507-40471

Figure 112. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Buna N, Minnesota Rubber Co., Compound 366 Y, Batch 982, Second Lot

Figure 113. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Buna N Nitrile, Parker Seal Co., Compound N741-75, Batch 132325, Second Lot

6507-40473

Figure 114. Log $\bar{P}$ vs $1/T(^{\circ}K)$ for H₂, Ar, Kr, and Xe for Silicone Rubber, Minnesota Rubber Co., Compound 71417, Second Lot

TABLE 51
COMPARISON OF PERMEABILITY ($\bar{P}$) OF BUNA N
(NITRILE) RUBBER SAMPLES A AND B

Gas	Temperature (°F) Sample Vendor	$\bar{P}$ (std cc-cm/sec-cm ² -cm Hg $\Delta P \times 10^{-9}$)							
		100		150		200		250	
		A	B	A	B	A	B	A	B
H ₂	MR	2.0	4.3	5.0	11.5	10.5	24	21.2	44
	PS	1.5	1.7	3.5	5.6	7.2	14	14.1	29
Ar	MR	0.5	0.23	1.25	1.1	2.6	4.0	5.2	9.5
	PS	0.25	0.24	0.54	0.8	1.0	1.9	1.8	4.0
Kr	MR	0.39	0.64	1.45	2.2	4.7	5.5	13.2	13.0
	PS	0.37	0.23	0.9	0.96	1.9	2.7	3.6	6.8
Xe	MR	0.47	0.66	1.75	2.6	5.2	6.6	13.4	16.0
	PS	0.43	0.14	1.1	0.68	2.4	2.2	4.9	6.0

MR = Minnesota Rubber Compound 366Y
PS = Parker Seal Compound N741

TABLE 52
COMPARISON OF PERMEABILITY ($\bar{P}$) OF SILICONE RUBBER
SAMPLES A AND B, MINNESOTA RUBBER
COMPOUND 71417

Temperature (°F) Sample Gas	$\bar{P}$ (std cc-cm/sec-cm ² -cm Hg $\Delta P \times 10^{-9}$)					
	100		200		300	
	A	B	A	B	A	B
H ₂	42	89	245	220	910	400
Ar	58	53	130	64	230	74
Kr	64	71	175	100	370	130
Xe	95	111	180	138	230	160

The solubilities of the test gases are obtained from the permeability tests. Table 53 summarizes the solubilities of H_2 , Ar, Kr, and Xe in 12 elastomers. Table 54 lists the solubility values for miscellaneous elastomer samples whose permeability has been measured, and a repeat of the Minnesota Rubber Buna N (366Y) sample. The agreement between the two samples of Buna N (366Y) rubber is quite good, except for the H_2 values which are widely separated. This tends to confirm the earlier supposition that material differences may be effecting the H_2 permeation rate.

A summary report on the permeation and solubility tests is being prepared.

D. GAS BOUNDARY LEAKAGE CALCULATIONS

The preliminary leakage calculations for the LMFBR-FFTF reactor cover gas containment system were completed. The calculated leakages are shown in Table 55 for individual components and for the complete assembly. These leakages are for reactor subassemblies exposed directly to cover gas during normal reactor operation, and assume 1.9% fuel failure. Not included are reactor service units, such as the CLEM, grapple adapters, and interim decay storage. Also not included are the inflation seal pressurization systems, buffer gas systems, and purge systems. There are no plans to calculate the leak rates of these systems at this time. No attempt was made to assess the effect of holdup time on leak rates. However, with neon (as an example), having a half-life of 38 sec, the anticipated holdup effect would significantly eliminate passage of radioactive neon particles.

The calculated krypton leak rate exceeds the allowable by a factor of 1.4, but the xenon leak rate is only 60% of the allowable rate. The hydrogen leak rate exceeds the allowable for neon by a factor of 840. Hydrogen was used in lieu of neon, per the seal test plan. (The similarity of the ionization potentials of neon and helium, the gas analyzer carrier gas, precluded the measurement

TABLE 53
SOLUBILITY OF GASES IN ELASTOMERIC MATERIALS

Elastomer	Vendor	Temperature Range (°F)	Gas	Solubility Range (cm ³ gas/cm ³ elastomer)
Buna N (N741)	Parker Seal	100-250	Ar	0.01-0.07
Buna N (366Y)	Minnesota Rubber	100-250	Ar	0.02-0.09
Buna N (969)	Greene Tweed	100-250	Ar	0.01-0.03
Urethane (P648)	Parker Seal	100-200	Ar	0.04-0.31
Urethane (FX522)	Minnesota Rubber	100-200	Ar	0.06-0.10
Urethane (379)	Greene Tweed	100-200	Ar	0.08-0.08
EPR (E692)	Parker Seal	100-300	Ar	0.11-0.14
EPR (559N)	Minnesota Rubber	100-300	Ar	0.05-0.10
EPR (952)	Greene Tweed	100-300	Ar	0.07-0.10
Silicone (S684)	Parker Seal	100-300	Ar	0.29-0.91
Silicone (71417)	Minnesota Rubber	100-300	Ar	0.38-0.52
Silicone (407)	Greene Tweed	100-300	Ar	0.28-0.82
Buna N (N741)	Parker Seal	100-250	H ₂	0.08-0.26
Buna N (366Y)	Minnesota Rubber	100-250	H ₂	0.09-0.24
Buna N (969)	Greene Tweed	100-250	H ₂	0.14-0.32
Urethane (P648)	Parker Seal	100-200	H ₂	0.03-0.25
Urethane (FX522)	Minnesota Rubber	100-200	H ₂	0.47-1.11
Urethane (379)	Greene Tweed	100-200	H ₂	0.27-0.37
EPR (E692)	Parker Seal	100-300	H ₂	0.31-0.44
EPR (559N)	Minnesota Rubber	100-300	H ₂	0.16-0.39
EPR (952)	Greene Tweed	100-300	H ₂	0.10-0.39
Silicone (S689)	Parker Seal	100-300	H ₂	1.05-3.29
Silicone (71417)	Minnesota Rubber	100-300	H ₂	0.70-1.62
Silicone (407)	Greene Tweed	100-300	H ₂	0.46-0.71
Buna N (N741)	Parker Seal	100-250	Kr	0.05-0.24
Buna N (366Y)	Minnesota Rubber	100-250	Kr	0.11-0.17
Buna N (969)	Greene Tweed	100-250	Kr	0.09-0.12
Urethane (P648)	Parker Seal	100-200	Kr	0.22-0.58
Urethane (FX522)	Minnesota Rubber	100-200	Kr	0.16-0.40
Urethane (379)	Greene Tweed	100-200	Kr	0.14-0.24
EPR (E692)	Parker Seal	100-300	Kr	0.12-0.24
EPR (559N)	Minnesota Rubber	100-300	Kr	0.12-0.20
EPR (952)	Greene Tweed	100-300	Kr	0.22-0.27
Silicone (S684)	Parker Seal	100-300	Kr	0.95-1.94
Silicone (71417)	Minnesota Rubber	100-300	Kr	0.86-1.74
Silicone (407)	Greene Tweed	100-300	Kr	0.29-0.88
Buna N (N741)	Parker Seal	100-250	Xe	0.14-0.36
Buna N (366Y)	Minnesota Rubber	100-250	Xe	0.25-0.46
Buna N (969)	Greene Tweed	100-250	Xe	0.20-0.27
Urethane (P648)	Parker Seal	100-200	Xe	0.33-0.51
Urethane (FX522)	Minnesota Rubber	100-200	Xe	0.24-0.56
Urethane (379)	Greene Tweed	100-200	Xe	0.24-0.50
EPR (E692)	Parker Seal	100-300	Xe	0.38-0.91
EPR (559N)	Minnesota Rubber	100-300	Xe	0.28-0.54
EPR (952)	Greene Tweed	100-300	Xe	0.22-0.38
Silicone (S684)	Parker Seal	100-300	Xe	1.55-3.53
Silicone (71417)	Minnesota Rubber	100-300	Xe	0.33-1.21
Silicone (407)	Greene Tweed	100-300	Xe	0.16-1.10

TABLE 54
SOLUBILITIES OF ELASTOMERS TO THE
TEST GASES H₂, Ar, Kr, AND Xe

Polyacrylic, A607		Buna S, G244		Polyacrylic, 335GA		Urethane, P642	
Parker Seal Company		Parker Seal Company		Minnesota Rubber Company		Parker Seal Company	
Temperature Range, 100 to 307°F		Temperature Range 101 to 251°F		Temperature Range 99 to 301°F		Temperature Range 103 to 202°F	
Gas	Solubility (cm ³ gas/cm ³ elastomer)	Gas	Solubility (cm ³ gas/cm ³ elastomer)	Gas	Solubility (cm ³ gas/cm ³ elastomer)	Gas	Solubility (cm ³ gas/cm ³ elastomer)
H ₂	0.97 to 1.53	H ₂	0.10 to 0.46		0.19 to 1.41		0.40 to
Ar	0.08 to 0.21	Ar	0.02 to 0.16	Ar	0.047 to 0.21	Ar	0.07 to 0.15
Kr	0.36 to 0.69	Kr	0.08 to 0.23	Kr	0.20 to 0.64	Kr	0.08 to 0.35
Xe	0.53 to 0.93	Xe	0.04 to 0.31	Xe	0.31 to 1.14	Xe	0.14 to 0.52
Butyl Rubber		Buna N (Nitrile) Rubber					
Parker Seal Company B 318-70		Minnesota Rubber Company 366Y, Batch 982, 2nd Lot		Parker Seal Company E259			
Temperature Range, 95 to 255°F		Temperature Range, 101 to 244°F		Temperature Range, 104 to 311°F			
Gas	Solubility (cm ³ gas/cm ³ elastomer)	Gas	Solubility (cm ³ gas/cm ³ elastomer)	Gas	Solubility (cm ³ gas/cm ³ elastomer)		
H ₂	0.29 to 0.69	H ₂	0.40 to 4.19	H ₂	0.60 to 0.85		
Ar	0.08 to 0.12	Ar	0.04 to 0.11	Ar	0.11 to 0.19		
Kr	0.14 to 0.23	Kr	0.18 to 0.22	Kr	0.07 to 0.43		
Xe	0.11 to 0.21	Xe	0.29 to 0.33	Xe	0.27 to 0.64		

TABLE 55
FFTF COVER GAS LEAK RATES

FFTF Component	Seals Per Unit		Units Per FFTF	FFTF Seal Leak Rate for Total Seals Noted [cc(STP)/sec] †				Metal Seal at Δ Argon = 0.47 psid	FFTF Total Leakage (Elastomer plus metal seals) [cc(STP)/sec] †			
	Elastomer	Metal		Elastomer Seal at ΔP = 29.92 in. Hg					Gas and Partial Pressure (atm)			
				Kr	H ₂	Xe	Ar		Kr 0.467-7	H ₂ 0.0025-7	Xe 2.54-7	Ar 0.086-10
IVHM Assembly	2	0	3	10.2-6 §	40.8-6 §	11.9-6 §	6.9-6 §		0.476-13 §	0.102-13 §	30.2-13 §	0.59-16 §
IVHM Arm Support	63	0	3	537.3	1600.0	840.0	430.0		250.9	4.00	2133.6	36.9
IVHM Orientation System	14	0	3	11.9	41.9	14.1	7.98		5.6	0.11	35.8	0.7
IVHM Chain Hoist	30	0	3	20.0	88.7	16.2	23.4		9.4	0.22	41.1	2.01
Fuel Transfer Port	4	0	3	0.36	0.84	0.36	0.09		0.17	0.002	0.91	0.008
Instrument Tree	20		3	78.6	362.1	94.2	69.0	0.163-6 §	36.7	0.90	239.2	5.9
		8							0.1	0.0004	0.414	0.01
Fuels Open Test Assembly	4		3*	1044.0	1983.0	146.0	375.0		487.0	4.96	2910.8	32.3
		2						0.141	0.1	0.0003	0.36	0.01
Control Rod Drive Mechanism Assembly	0	4	18					37.8	17.7	0.09	96.0	3.25
Temperature and Liquid Level Port Plug and Instrumentation	0	4	3					0.133	0.06	0.0003	0.34	0.011
Closed Loop In-Reactor Assembly	0	1	3*					0.063	0.03	0.0002	0.16	0.005
Proximity Test Plug	0	2	3*						0.002	0.00001	0.01	0.0004
Low Level Flux Monitor	0	1	3					0.117	0.05	0.0003	0.30	0.01
Closure Head Assembly	0	2	1					97.6	45.5	0.24	248.0	8.4
									853.8	10.38	5753.2	84.3
									or	or	or	or
									8.5-11	1.0-12	5.8-10	8.4-15
									6.0-11	Ne = 8.4-14	9.6-10	2.4-4
Allowable FFTF leakage												

*The OTA, CLIRA and Proximity Test Plug Ports total 9

†STP is standard atmosphere (29.92 in. Hg) and temperature (0 °C)

§10.2-6 means 10.2 x 10⁻⁶

of neon.) Limited information in the literature shows hydrogen to have twice the permeation rate of neon, through silicone rubber at 0°C.

The outstanding sources of leakage in the system occur through a set of EPR seals, operating at 167°F in the arm support subassembly of the IVHM, and through a set of silicone seals, operating at 300°F in the materials open-test assembly. The large silicone shaped seal on the arm support system, and the inflatable seals on both the arm support system and instrument tree are part of a system of seals in parallel and series during normal reactor operation. As a result, their relatively high leak rates are reduced by other seals in the same leak paths. Consequently, the total effective leakage of these seals is low.

A report^{*} was issued which describes the method of calculating and tabulating all of the data used to calculate the leak rates.

E. INSTRUMENT TREE INFLATABLE SEALS TEST

The test rig fabrication was completed. The silicone seals are currently scheduled for delivery to AI in July 1974, and the EPDM seals have been promised for September 1974 delivery. Westinghouse comments on the Test Plan are being incorporated in a revision to the Test Plan.[†]

F. COMPARATIVE LEAK RATE

To establish the reliability of seal leakage data, a test was planned to measure the gaseous permeation and leakages through an elastomeric seal and a metallic seal, independently by AI and HEDL. Upon completion of the first phase of tests, the fixtures would be shipped to the alternate laboratory. The measurements would then be repeated and the results compared.

A test fixture (Figure 68) was fabricated at AI. A test procedure[§] was released.

*"FFTF Cover Gas Leakage Calculations," TI-707-810-014, June 17, 1974

†"FFTF Instrument Tree Inflatable Seal Test Plan," TP-707-810-007, February 6, 1974

§"Cover Gas Seal Development: AI/HEDL Comparative Test — Static Elastomer and Metal Seals," DTP-707-810-006, February 14, 1974

The elastomer seal test, using Buna N O-rings (Minnesota Rubber, Compound 366Y) was set up and conducted for 1507 hours at $70 \pm 5^\circ\text{F}$. The measured leakage of H_2 , L_{H_2} , was 11.0×10^{-8} std/cc/sec/linear in. of seal/atm ΔP . Using the 6.7 Kr equivalent value for H_2 under these conditions, the L_{Kr} value is 1.64×10^{-8} as compared to an L_{Kr} value of 0.9×10^{-8} calculated from permeability and geometry factors.

During the test, it was observed that a total of only about 0.025 cc of H_2 permeated through the first O-ring seal, and only the barest trace of H_2 (too low to measure) was found permeating through to the second seal. No Kr gas was found permeating through either seal, although small concentrations (10 ppm or less) of Kr are difficult to detect because of the closeness of the N_2 and Kr chromatographic traces.

The HEDL test fixture was received and installed in the test fixture, (Figure 115) and the testing started. The initial reading at 70 hours indicated a total seal leak rate of about 8×10^{-8} std cc/sec of H_2 with a pressure differential of 39.5 psig across the seal. The testing of the metal seal was stopped when it was apparent that a significant amount of gas was leaking from the sampling reservoir through the silicone rubber O-rings. Permission was given by HEDL personnel to attempt to seal the system (externally) to prevent this loss. Without disturbing the metal seal, Buna N O-rings were forced into the gap between the top and bottom portions of the test fixture, and the closure bolts and spacers were sealed with a RTV silicone cement.

Testing was resumed and a fairly linear leak rate was obtained. The test was continued for 722 hours. Examination of the data indicated that the temporary fixture seal was leaking. A preliminary estimation of the leak rate is 5.7×10^{-8} cc/sec of H_2 gas at a 39.5 psig differential. Assuming that the Ar and H_2 exhibit similar flow characteristics, and reducing the pressure differential to 1 atm, the approximate total leak rate is 1.4×10^{-6} cc/sec/atm ΔP .

G. SEALS IN SODIUM VAPOR

A Test Plan* was written outlining a test of reciprocating Quad, "Q" and "T" elastomer seals exposed to a sodium vapor environment found in a "severe

*"Plan for Testing of Elastomer Seals in Sodium/Sodium Vapor for FFTF Applications," TP-707-250-001, October 31, 1973

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Figure 115. AI-HEDL Comparative Seal Test Apparatus
With HEDL Metal Seal Fixture Installed

typical" case in the FFTF head penetrations. The test conditions would duplicate the reciprocating seal conditions in Section III-A above except that the seals are 1 in. in diameter and they are exposed to sodium vapor.

The test fixture and P&I diagram for installation in a sodium loop was designed and procurement of materials initiated for fabrication. A shifting of priorities in LMFBR, however, resulted in stopping further effort this fiscal year.

H. IVHM INFLATABLE SEAL

1. Completion of Test Program

All testing except the post-test permeation measurement was conducted in FY 73 and has been reported.*

In this reporting year the IVHM post-test measurement of permeation through the inflatable seals was conducted, using 1% H₂ and 1% Xe trace gases in argon. The inflation pressure was 15 psig, the differential pressure was 2.5 psi, the temperature was 150°F, and the test duration was 384 hr. A gas chromatograph was used to measure the permeation of the trace gases through the seals.

Permeation leakage through the elastomer seal can be expressed by

$$R = \bar{P} \times K_{H/T} \times L \times \Delta P \quad \dots(1)$$

where

R = permeation rate (cc/sec)

$\bar{P}$ = permeation coefficient of the elastomer (cc-cm/sec-cm²-atm)

K_{H/T} = cross section coefficient of the seal (cm/cm)

L = length of seal (cm)

ΔP = differential partial pressure of the gas (atm).

The K_{H/T} value was determined, using the R and ΔP observed during this test, along with the measured seal length (L) and the permeation coefficient measured

*"Annual Technical Progress Report, Sodium Technology and Cover Gas Seal Development Programs Government Fiscal Year 1973," AI-AEC-13110, September 15, 1973

previously^{*}. With the $K_{H/T}$ determined, permeation of the various reactor cover gas species can be determined, using the gas specie partial pressure and $\bar{P}$. The values of $K_{H/T}$ determined are:

$$K_{H/T} = 1.42, \text{ hydrogen basis}$$

$$K_{H/T} = 2.30, \text{ xenon basis.}$$

While, ideally, these values should be identical, the agreement is good, and well within the expected experimental error. An AI Test Report[†] was issued covering the entire test program.

2. Adhesive Failure Evaluation

Following the completion of the formal IVHM Rotating Plug Seal Test, a special test, requested by Westinghouse personnel, was conducted to support troubleshooting of some IVHM problems at HEDL. The seal was operated at normal conditions of 100°F, 2 ft/min, with 8-psig inflation pressure. The torque recorder showed chatter; and, after 1.5 revolutions, the inflation pressure of the larger-diameter seal dropped to zero. The torque was within the normal range of 32 to 42 lb/ft. Examination of the seal revealed that the rubber was torn at the stem, due to movement of the seal with respect to the seal housing (Figure 116). This movement occurred because of a breakdown in the adhesive bond between the seal and seal gland. Failure Report No. 309 was issued. The mode of failure was very similar to the failure of the smaller-diameter seal, as noted in Failure Report No. 280, dated April 9, 1973, in which MEK solvent used in a rubber sealant for the test apparatus was suspected of degrading the effectiveness of the cement used to bond the seal to the steel housing.

A review of the materials involved in the seals installation and testing was conducted. The following listing summarizes the review:

- 1) The seal material is B. F. Goodrich Nitrile No. 139-PC-12.
- 2) The seal was installed per AI Specification ST0620NA0087, which calls for cleaning the seal with methyl ethyl ketone (MEK) and the

^{*}AI-AEC-13110, page 144, for B.F. Goodrich Nitrile No. 139-PC-12

[†]"Test Report Development IVHM Reactor Refueling Plug Dynamic Seal Test," TR-707-810-004, May 8, 1974

Figure 116. IVHM Development Test Inflatable Seal — Adhesive Condition

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gland with denatured alcohol. Dow Silastic 140 RTV, a silicone-base adhesive, was used to cement the seal in the gland. Dow 705 silicone oil was used as original lubricant, but was absorbed by the rubber after 1 week, and Dow Corning 55M grease was applied, per the "A" change of the specification.

- 3) A leaky gasket in the test apparatus was plugged, using a liter of liquid rubber sealant with 50% MEK solvent. Some air drying was allowed. Condensed MEK was observed in tygon tubing to the inner cavity later.
- 4) The gasket was later removed and replaced with Permatex 2F which is a sealant-adhesive with 5% isopropyl alcohol in it. The Permatex air dried for a few days before closing the test rig.

A series of compatibility and adhesive-bond strength tests was started, to evaluate materials in the test setup and materials that are backup candidates. Reference materials are B. F. Goodrich Buna N 139-PC-12 rubber, 140 RTV adhesive, and DC55M lubricant. Backup materials are Chemlock 304 adhesive and Exxon WSX8788 lubricant. The effect of Permatex 2F sealant and MEK used in the test rig, plus the effect of Dow 705 on the bond, is also being evaluated.

To summarize the test results to date, the following have been observed:

a. Compatibility Tests

After the inner seal failure, a section of seal bonded to a steel plate with 140 RTV was exposed to saturated MEK vapors for 2 weeks at 150°F. The seal had delaminated and fallen off the plate. The adhesive was also dissolved. This was thought to be the cause of failure until the outer seal failed nine months later. The outer seal would see only those MEK vapors that would diffuse through the inner seal.

Pieces of 140 RTV and seal rubber were immersed in DC55M grease and Dow 705 oil and left in air at 165°F. Periodically they were removed, wiped off and weighed (Table-56). The 140 RTV increased weight by 35% in DC55M after 90 days and 0% in Dow 705 after 60 days. The rubber lost weight by 8% after 90 days in DC55M and 12% after 90 days in air. This implies that the rubber in DC55M absorbed 4% DC55M while losing rubber plasticizers up to 12%.

TABLE 56
RESULTS OF WEIGHT CHANGE TESTS ON
SEAL ASSEMBLY MATERIALS

Soak Time (days)		10	20	60	90	180
Specimen	Lubricant	% Wt Change	% Wt Change	% Wt Change	% Wt Change	% Wt Change
140 RTV	DC55M	+8.1	+32.0	+29.0	+35.0	28.4
140 RTV	WSX 8788	+2.0	+2.0	+ 1.5	+ 1.6	1.6
140 RTV	Glycerin		+2.0	- 2.8	+ 2.9	
140 RTV	Dow 705	+1.0	0.0	0.0	0.0	0.0
140 RTV	None	-0.4	-0.5	- 0.5	+ 0.5*	0.0
Chemlock 304	DC55M	+1.5	+3.2	+ 3.6(48)†	+4.4(126)	4.5
Chemlock 304	WSX 8788	+1.3	+1.8	+ 2.2(48)	+2.5(126)	2.4
Chemlock 304	Glycerin		+4.0	5.4	5.4	
Chemlock 304	None	-0.03	-0.03	- 0.02(46)	+ 0.4*	1.2
B. F. G. Rubber	DC55M	-4.6	-6.8	-10.0	- 8.0	-8.2
B. F. G. Rubber	WSX 8788	-8.5	-9.1	- 9.0	- 9.0	-7.9
B. F. G. Rubber	Glycerin		+3.5	+ 4.9	-12.0*	-13.3
B. F. G. Rubber	None	-11.0	-12.0			

*Possibly moisture was absorbed during cooldown (rainy weather)

Strips of adhesive (140 RTV or Chemlock 304) or rubber were smeared with, or dipped into lubricant and held at 165°F in air even for time indicated.

†() Actual days if different from column heading

b. Pull Tests*

Sections of seal were bonded to stainless-steel plates with 140 RTV, smeared with either DC55M, Dow 705 oil or left dry. They were then held at 165°F for various times, removed and pull-tested for shear strength (Table 57 and Figures 117 and 118). The sections smeared with DC55M showed no definite pattern of weakening after 6 weeks exposure, and had strength a little less than the ones in air. The samples with Dow 705 oil had the same strength as the ones in air after 9 weeks. Longer term tests are in process.

A test similar to 2-a (above) using only 2 oz of MEK in a cannister with a cover taped shut, was held at 165°F for 2 weeks. The bond was good and a pull test showed a shear strength of 34 psi, which is slightly less than for samples heated in air.

Another test similar to 2-a (above) using a cup of Permatex 2F in a cannister with a cover taped shut, was held at 165°F for up to 6 weeks. The 2-week samples had a shear strength of 14 psi, while the 6-week samples had a 44-psi shear strength. The isopropyl alcohol vapors had been driven out of the cannister at 6 weeks.

c. Preliminary Conclusions

- 1) The MEK may have caused or contributed to failure of the inner seal bond, but not necessarily to failure of the outer seal bond which saw only those vapors that permeated through the inner seal.
- 2) It is doubtful that Dow 750 oil or the small amount of isopropyl alcohol in the Permatex did any damage.
- 3) The DC55M grease could well have caused excessive swelling of the 140 RTV and destroyed the bond after 3300 hours exposure at 150°F.

It should be noted that there was a 35% weight increase of 140 RTV soaked in DC55M for 90 days. The mechanism for grease getting to the adhesive is not clear but considerable grease was on the seals and could have been transferred by a wicking or capillary action between the seal and the gland walls.

*Note: Normal operation of the IVHM applies a 2-psi shear stress to the adhesive

TABLE 57

RESULTS OF PULL TESTS ON IVHM SEAL-ADHESIVE-METAL BOND
 (All specimens soaked at 165°F and pulled at room temperature)
 (Specimens smeared with lubricant as indicated)

Adhesive	Lubricant	Test Results											
140 RTV	DC55M	Specimen No.	1	12 [*]	24	33	34	2	13 [*]	25	26 [*]	27 [*]	
		Soak Time (weeks)	2	2	2	2	2	6	5	6	3	6	
		Shear Stress (psi)	25	44	63	59	62	28	34	32	20	12 [§]	
140 RTV	WSX 8788	Specimen No.	35	8	16 [*]	36	9	17 [*]					
		Time (weeks)	2	3	3	4	8	12					
		Stress (psi)	70	17	49	39	18	41					
140 RTV	None	Specimen No.	20 [*]	30	31	21 [*]	X						
		Time (weeks)	2	2	4	6	†						
		Stress (psi)	60	73	>78	55	42						
140 RTV	Dow 705 Oil	Specimen No.	A	B	C	D	E	F					
		Time (weeks)	1.5	3.0	4	9	13	18					
		Stress (psi)	55	47	82	40	61	48					
Chemlock 304	DC55M	Specimen No.	5	14 [*]	6	15 [*]							
		Time (weeks)	3	3	9	12							
		Stress (psi)	80	>109	>73	>66							
Chemlock 304	WSX 8788	Specimen No.	10	18 [*]	11	19 [*]							
		Time (weeks)	3	3	8	12							
		Stress (psi)	>113	>90	124	>71							
Chemlock 304	None	Specimen No.	7	22 [*]	23 [*]								
		Time (weeks)	1 day	3	12								
		Stress (psi)	>50	>105	>72								
140 RTV in can with 1 cup Permatex 2F		Specimen No.	3	28	29	4							
		Time (weeks)	2	2	4	6							
		Stress (psi)	14	15	65	44							
140 RTV in can with 2 oz MEK		Specimen No.	32	MEK had fully evaporated and diffused out of can through cover									
		Time (weeks)	2										
		Stress (psi)	34										

>xx Rubber tore before adhesive sheared

X No specimen number assigned

*No cleaning with MEK during installation

†Set 4 months at room temperature with no lubrication

§Very little bond area. Poor bond.

Samples prepared in groups at three times:

Group I, Specimens 1 to 11

Group II, Specimens 12 to 23

Group III, Specimens 24 to 36

Group IV, Specimens A to F

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Figure 117. Pull Test Coupon (Section of IVHM inflatable seal, cemented to Type 304 stainless steel with Dow Corning Silastic 140 RTV adhesive)

Figure 118. Pull Test Apparatus

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Some diffusion through the rubber is also possible. It is noteworthy that the first failure occurred after 4 months from installation and the second one 9 months later. During the 9 months there were no operations of the test rig. A replacement seal was installed for the inner seal 2 months before the outer seal failure and a 384-hour permeation test was run at 150°F with 15-psig inflation pressure. The first rotation after that test resulted in failure of the outer seal. Pull test sections of seal sections cemented with 140 RTV to stainless steel and smeared with DC55M have been put in 150°F ovens for long term exposure of several months and greater. The tests reported in Table 57 were only 6 weeks long and may have been too short to duplicate the IVHM seal adhesive failure.

I. CRBRP ROTATING PLUG SEAL FEATURE TEST

This program consists of the evaluation of lubricants for inflatable seals, development of procedures and issuance of a specification for installation and removal of a seal, conducting long-term adhesive bond tests, and conducting a test on the IVHM seal with CRBRP seal conditions.

1. Lubricant Evaluation

The inflatable seals used in the Clinch River Breeder Reactor Program (CRBRP), operate at higher speed, higher inflation pressures, and for longer total travel than the previously qualified IVHM seals.* A program was conducted to evaluate several candidate lubricants at the CRBRP conditions. Greases that had been evaluated at their maximum use temperature requirements were re-evaluated at the 100 to 150°F temperature range of CRBRP reactor use. A series of compatibility and lubricity-wear tests were also performed.

Based on these tests, DC55M was recommended for use on the CRBRP seal because of low friction and wear, good thermal stability, good compatibility with the seal rubber and Chemlok 304 adhesive, ready availability and favorable operating experience with inflatable seals and with other seals in the LMFBR general seal test program.

*"Plan for Testing the IVHM Inflatable Seal to CRBRP Conditions," TP-707-810-008, February 11, 1974

a. Compatibility Tests

Compatibility or weight change tests of seal materials and adhesives with various lubricants at 100 and 150°F are being conducted. Also, lubricant weight changes at those temperatures are being observed. Tables 58, 59 and 60 show the results to date and indicate future data points. Originally, tests of this type were conducted at 165°F to accelerate effects (Table 56). The 100 and 150°F tests give data within the range that inflatable seals will operate. The 140 RTV is in the matrix as it is still used on the IVHM seal. Glycerin was a candidate lubricant when the test was started but has since been dropped from consideration based primarily on its high evaporation and high starting friction. The Chemlok 304 shows low absorption of the lubricants while 140 RTV absorbs considerable DC55M at all the temperatures.

TABLE 58
RESULTS OF WEIGHT CHANGE TEST ON SEAL ASSEMBLY
MATERIALS Soaked at 100°F in Various Lubricants

Soak Time Days		10	20	45	60	90
Material	Lubricant	Percent Weight Change				
140RTV	DC55M	14.5	31.6	37.8	38.5	38
140RTV	WSX8788	1.0	0.9	0.6	0.6	0.7
140RTV	Glycerin	-0.2	-0.2	-0.1	+0.1	0.0
Chemlok 304	DC55M	0.3	0.6	0.9	1.2	1.5
Chemlok 304	WSX8788	0.4	0.7	0.8	0.9	1.0
Chemlok 304	Glycerin	-0.1	-0.1	0.1	0.4	0.3
BFG Rubber	DC55M	-1.2	-2.8	-4.3	-5.2	-6.6
BFG Rubber	WSX8788	-4.0	-5.6	-6.8	-7.5	-8.6
BFG Rubber	Glycerin	0.3	0.6	0.9	0.8	0.8

TABLE 59
RESULTS OF WEIGHT CHANGE TEST ON SEAL ASSEMBLY
MATERIALS Soaked at 150°F in Various Lubricants

Soak Time Days		10	20	45	60	90
Material	Lubricant	Percent Weight Change				
140RTV	DC55M	17.8	37.0	39.6	40.3	41
140RTV	WSX8788	0.9	0.2	-0.9	-1.3	-0.2
140RTV	Glycerin	-0.5	-0.5	-0.2	-0.2	-0.7
Chemlok 304	DC55M	1.1	1.9	2.5	2.7	3.3
Chemlok 304	WSX8788	0.5	0.9	1.3	1.5	1.6
Chemlok 304	Glycerin	0.5	1.0	1.9	2.4	2.8
BFG Rubber	DC55M	-2.0	-5.9	-8.3	-9.3	-10.8
BFG Rubber	WSX8788	-6.0	-9.0	-10.3	-10.4	-9.6
BFG Rubber	Glycerin	0.8	0.4	-1.8	-2.3	-3.3

TABLE 60
WEIGHT CHANGE OF SEAL LUBRICANTS
DURING THERMAL SOAKING IN AIR

Lubricant	Temp (°F)	Percent Weight Change				
		Soak Time, Days				
		10	20	45	60	90
WSX8788	100	-0.2	-0.2	-0.3	-0.1	-0.1
WSX8788	150	-0.1	-0.1	-0.1	-0.1	-0.2
DC55M	100	-0.1	-0.1	-0.1	-0.1	-0.3
DC55M	150	-0.1	-0.1	-0.1	-0.2	-0.2
Glycerin	100	-2.2	-2.4	-0.1*	-3.3†	-5.8
Glycerin	150	-6.8	-8.2	-9.3*	-24.1†	-29.5
Glycerin	165	-37.0	-38.0	-6.5*	-77.0†	-79.0

*30 days

†75 days

b. Lubricity and Wear Testing

The wear and lubricity tests in support of the CRBRP seal use has been completed and is summarized in Table 61. Note that DC55M was the only grease that did not experience a shearing of the overload pin on the test fixture during startup after a dwell. The test rig used is the one described in the lubrication evaluation in an earlier section of this report.

2. Seal Installation and Removal Using Chemlok 304 Adhesive

When the 140 RTV adhesive used to bond the IVHM to its gland exhibited failures, Chemlok 304 was selected as a backup material because of its good compatability with seal lubricants (Table 56) and high shear strength (Table 57). With this selection, it became necessary to develop installation and removal procedures and processes, and to draft them into a process specification. The removal procedures were first developed using flat plates and seal sections. These procedures were demonstrated using a straight 3-ft section of seal gland machined for this use. Figure 119 shows the operation which involves cutting a window in the seal, inserting a pry bar, lifting the seal, cutting clean through it and then separating the adhesive-metal band with a putty knife while prying the seal up. Residual Chemlok 304 can be softened with Oakite Stripper 157 and scraped from the metal. Specification ST0620NA0095, describing the process in detail, is being prepared.

The installation procedure using Chemlok 304 was developed and tried on the 3-ft gland section and then used to install the seals in the test rig. It, too, is described in the above specification being prepared. The steps are summarized as follows and as shown in Figures 120 through 125.

- 1) Mix 250 gm of the 2-part Chemlok 304 adhesive, put it into a squeeze tube, attach the extruding key, flatten the tube nozzle and weigh the tube assembly, plus gauze to be used for cleanup (Figure 120).
- 2) Extrude an even band of adhesive into the gland and spread, using the spreader tool. Keep adhesive away from the inflation port but put some around the inflation stem (Figures 121 through 123).
- 3) Pick the seal up, insert the stem into the port and lay the entire seal in the gland (Figure 124).

TABLE 61

CRBRP LUBRICANT WEAR EVALUATION FOR INFLATABLE SEALS
 (Nitrile O-Rings, 4 in. ID x 0.210 in. Cross Section,
 50 fpm at 150°F, 10% Squeeze on O-Rings,
 80,000-ft life travel minimum)

Lubricant	Travel (ft)	Drag (lb/in.)	O-Ring		
			Weight Change	Radial ΔD , in.	Longitudinal ΔD , in.
WSX8788, W/5% MoS ₂	60,000	<4	+0.7%	Negligible	+0.006
DC55M, W/5% MoS ₂	103,000	<2.5	Negligible	+0.002	+0.002
MoS ₂ Bonded Dry Lube (Perma Slick) 0.0007 in.	1,025	2 to shear of pin ⁽¹⁾	+0.2%	Negligible	Negligible
Glycerin Test 1	10,300	<2.0 ⁽²⁾	Negligible	+0.001	+0.002
Glycerin Test 2	86,355	2-3.5 ⁽³⁾	Negligible	-0.001	+0.001
WSX8788 1	87,000	<2.0 ⁽⁴⁾	+0.5%	Negligible	+0.001
DC55M 1	84,100	<2.0 ⁽⁵⁾	+0.3%	Negligible	-0.001
WSX8788 2	90,000	<2.4 ⁽⁶⁾	+0.7%	+0.001	+0.001
DC55M 2	85,200	<1.9 ⁽⁷⁾	+0.6%	Negligible	+0.001

(1) A pin shears at 8 lb/in. drag.

(2) Stopped over weekend. Pin sheared at 70°F on Monday.

(3) Stopped for 2.5 hr. Pin sheared at 80°F.

(4) Left at room temperature overnight and pin sheared when restarted.

(5) Left at room temperature for five hours and restarted with 2.0 lb/in. drag. Then left for weekend (65 hr) and restarted with 2.4 lb/in. drag.

(6) Left at 68°F overnight and 2 five minute dwell. Sheared pin each time.

(7) Left at 150°F for 5 min dwell and restarted with 2.4 lb/in. drag then at 70°F overnight and 2 five minute dwells with 2.4 lb/in. starting drag.

a. Starting
Cutout
←

9006-40191A

b. Cutout
Complete
←

9006-40190A

9006-40188A

c. Cutting Through Seal

9006-40187A

d. Removing Seal with Putty Knife-Pry Bar

Figure 119. Removal of Epoxy-Bonded Inflatable Seal

Figure 120. Adhesive Preparation

9006-40195A

Figure 121. Adhesive Application

9006-40196A

Figure 122. Adhesive Spreading

9006-40197A

Figure 123. Adhesive Application in
Inflation Stem Region

9006-40198A

Figure 124. Seal Inflation Stem Insertion 9006-40200A

Figure 125. Use of Seal Seating Roller 9006-40203A

- 4) Using the wide seal-seating roller, press the seal down to seat the seal under the retaining beads (Figure 125). Start at the inflation stem and seat 3 to 4 in. at a time followed by rolling with a 3-in. -diam by 1/4-in. wide rounded roller, not shown, to press the center of the seal base against the adhesive and to force air out. Proceed around the whole seal in this manner.
- 5) Weigh the tube assembly and gauze with wipings. Use $135 \text{ gm} \pm 15 \text{ gm}$ to give a 0.030-in. -thick adhesive layer.

3. Seal Removal and Cleanup Development Using 140 RTV Adhesive

During preparation of the test rig for the CRBRP test, the inner seal which was bonded in the gland with 140 RTV was mistakenly removed from the gland. The peel-pull effort to remove the seal was 10 to 20 lb. The inflation stem flange (molded into the seal) was bent 15 degrees during removal. One superficial scratch on the side of the seal was noted. This removal action prompted development of a seal and gland cleanup procedure to prepare for reinstallation of the seal.

Removal of 140 RTV from the seal or gland is accomplished with Cee-Bee C105a supplied by Cee Bee Chemical Co. It is an aliphatic petroleum and base solvent with no halogens. An overnight soak of a seal section with adhesive in the solvent resulted in dissolution of the adhesive and no damage to the seal.

Removal of the DC55M grease can be done using John B. Moore Corp. solvent M-6 or M-114. Short-time wiping only should be done to the seal as extended soaking can cause attack of nitrile rubber.

Cleaning of the seal removed from the test rig and of the gland was demonstrated using the above solvents. Details of the procedure are being documented in a specification to be issued.*

Pull-test samples, using sections of seal and sections of stainless-steel plate from which 140 RTV had been cleaned, were made up using Chemlok 304 adhesive to see whether the cleanup affected the bond. One was pulled after a 3-day cure and one after a week at 150 °F. The rubber started to tear in both cases at greater than 65-psi shear stress at the cement bond. The cleanup showed no effect on the bond.

*"Seal, Dynamic Inflatable, IVHM-RRP, Installation and Removal of," ST062-NA0096, July 18, 1974

4. CRBRP Test Operation

The AI Test Plan (Westinghouse TP 32.06) was revised to incorporate comments by Westinghouse and was reissued. A development test procedure for use by AI laboratory personnel was written and issued.

The inner seal inflation stem flange that was bent during seal removal was straightened to an 8-degree bend and the seal was installed in the test rig with Chemlok 304 following the procedures developed on the 3-ft test section. The tubular seating press was used and required considerable effort (~130 lb) to push the seal below the retaining ridges in the gland. Also, a hydraulic locking and bubble trapping effect was noted to occur if each 3- to 4-in. section was not fully seated before proceeding to the next 3- to 4-in. section. About 158 gm of adhesive for 19 ft of seal was used which should result in 0.036-in.-thick adhesive. The adhesive has in excess of a 2-hr pot life. The seating required 1 hr with 2 men working.

A roller type seating tool was developed using the 3-ft seal gland test section and some scrap seal sections. The amount of adhesive also was varied, and less adhesive resulted in less hydraulic lock while still providing a full bond area.

The outer seal was installed using the seal seating roller and less cement. The upper edges of the retaining ridge in the gland were lightly dabbed with adhesive to provide lubrication during seating. A total of 134 gm of adhesive for 19 ft of seal was used which results in 0.028-in.-thick adhesive. One man seated the seal in 1/2 hr. The process is documented in a specification.*

After a 3-day cure, the seals were lubricated with DC55M; 57 gm was applied to the outer seal and 27 gm to the inner seal to evaluate lubricant effectiveness as a function of quantity. The top cover was put in place and tilted to build in ± 0.060 in. of vertical runout.

The non-uniform rotation cycle (1-1/2 CW, 1 CCW) required buildup of a timing switch box which is now installed. The run-in cycles performed and torque values compared well with previous test results. The installation was leak checked, brought up to 150°F, started into tests which vary speed and

*"Seal, Dynamic, Development Test, Installation and Removal of," ST062NA0095, July 18, 1974

inflation pressure. Life cycling to 80,000 ft of total travel will be conducted followed by an examination of the seals when additional tests will be defined.

An order was placed for a spare set of seals to have on hand if needed to replace the seals in test.

5. Seal Material and Configuration

The supplier of the IVHM Inflatable Seal, B. F. Goodrich, was contacted for information on the aging characteristics of the 139-PC-12 nitrile compound used for the seal. The compound has additives for ozone and oxidation resistance and is also their best nitrile for radiation resistance. Nitrile is generally noted for poor aging. Compounding it with polyvinyl chloride (PVC) rubber gives excellent aging qualities but chlorides are not permitted per RDT spec M 11-1T.

B. F. Goodrich was sent sketches (Figure 126) of designs of an inflatable seal configuration which would provide a mechanical interlock to supplement the adhesive. They will comment and/or add to the design concepts.

6. Adhesive Performance Tests

Initial bond strength performance tests of pull test samples are shown in Table 57. Test samples and equipment are shown in Figures 116 and 117. The high strength shown added to the decision to use Chemlok 304 for CRBRP but additional performance data are being generated.

Additional bond strength tests were started. Pull-test specimens were prepared using sections of IVHM inflatable seal cemented with Chemlok 304 to stainless-steel and carbon-steel plates. Specimens were smeared with lubricants WSX8788 or DC55M, and are being soaked in air at 150°F for up to 16 months. At various times specimens will be pull-tested to note the effect of time, temperature, and lubricant on the adhesive bond. Results to date are shown in Table 62. All shear strengths are high and acceptable. The lower values for stainless-steel substrates are probably a surface finish effect, as those samples used rolled plate stock with about a 16 μ -in. finish.

RUBBER COMPRESSED AT LIP
KNURL OR ROUGHEN METAL SURFACES
USE WITH ADHESIVE AS PRIMARY HOLDDOWN

9006-40174

a. Approach 1

MOLD BOTH SEALS INTEGRALLY OR VULCANIZE A
CONNECTING STRIP TO BOTH SEALS AFTER EACH
IS MADE
USE WITH ADHESIVE AS PRIMARY HOLDDOWN

9006 40175

b. Approach 2

1/2 in. DIAMETER STUBS OF RUBBER AND MATCHING HOLES
IN BASE AT 6 in. INTERVALS
USE WITH ADHESIVE AS PRIMARY HOLDDOWN

9006-40176

c. Approach 3

3/16 in. DIAMETER STUBS OF METAL AND MATCHING HOLES
IN BASE AT 6 in. INTERVALS
USE WITH ADHESIVE AS PRIMARY HOLDDOWN

9006-40176A

d. Approach 4

Figure 126. Mechanical Holddown for Inflatable Seals

TABLE 62
PULL TESTS ON CRBRP SEAL-CHEMLOK ADHESIVE-METAL
BOND WITH LUBRICANT AND TIME SHOWN
AT 150°F IN AIR

Substrate	Lubricant	Time (mo)	Shear Strength (psi)
Stainless Steel	DC55M	1	96
Stainless Steel	DC55M	2	92
Carbon Steel	DC55M	2	>120
Stainless Steel	WSX8788	1	164
Stainless Steel	WSX8788	2	63
Carbon Steel	WSX8788	2	>164

>Means rubber tore and pull stopped

J. TOP SHIELD DIP SEAL PARAMETRIC TEST

The revised Test Plan for this subtask* was written and submitted to ARD for review and comments.

Design and fabrication of the preliminary, water test fixture (transparent dip seal) was completed and testing begun.

Design of the dip seal — annulus test fixture was initiated. The test fixture as currently designed will have a nonrotating blade, but with some change, could be made into a system with a rotatable blade, if required. The annulus gap will be 1/2 in. and the circumferential length will be approximately 12 ft.

An analytical study of the rate at which sodium oxide (Na_2O) deposits on annulus and dip seal walls would be dissolved and removed by sodium flushing was completed. Boundary conditions assumed included: (1) both walls at equal and constant temperature, (2) oxide deposit on both walls, and (3) laminar flow. Figure 127 shows predicted local sodium oxide dissolution rates for the case of 1/2 in. wide channels. In this figure, $\underline{a}$ is the ratio of the deposited oxide surface area to the projected area, $\underline{m}_i$ is the initial mass fraction of oxygen in the sodium, and $\underline{x}$ is the distance downstream from the starting point of the deposit. It is seen in Figure 127 that the oxide dissolution rate increases with flow rate and temperature, and decreases with distance downstream from the starting point of the deposit. Also shown in Figure 127 is Reynolds number.

*"Test Plan for the Dip Seal and Upper Annulus Sodium Deposition Test,"
TP-707-230-002, April 10, 1974

3507-40476

Figure 127. Sodium Oxide (Na_2O) Dissolution Rate in 0.5-in.-Wide Channel

An analytical study of transport of radioactive gases through the dip seal was initiated, and a report written describing this study.*

III. IMPACT ON LMFBR PROGRAMS

The initial phases of this program have guided selection of materials and seals for the FFTF and CRBRP from information on permeability, compression set and gland design published in progress and technical reports. FFTF designs of the IVHM inflatable and primary seals were verified during testing. The later phases will verify the instrument tree inflatable seal design and evaluate the use of inflatable seals for CRBRP. Continued testing of materials and seals will give confidence to the data already collected, define long-term performance, and develop materials and seals for longer term use. Specifications and a design guide for use in the LMFBR industry will become available.

*"Dissolution of Na_2O Deposits by Flushing with Sodium," TI-707-230-009, May 30, 1974

IV. SCHEDULE

The program schedule below shows the activities that were performed in FY 1974 and updates the planned tasks to be carried out in FY 1975.

Task 11 - FFTF-LMFBR Seal Test Program, Schedule and Milestones