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Performance Testing of Elastomeric Seal Materials Under Low- and High-Temperature Conditions: Final Report

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PERFORMANCE TESTING OF ELASTOMERIC SEAL MATERIALS UNDER LOW- AND HIGH-TEMPERATURE CONDITIONS- FINAL REPORT

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ABSTRACT

The U.S. Department of Energy Offices of Defense Programs and Civilian Radioactive Waste Management jointly sponsored a program to evaluate elastomeric O-ring seal materials for radioactive material shipping containers. This report presents the results of low- and high-temperature tests conducted on 27 common elastomeric compounds.

ACKNOWLEDGMENTS

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EXECUTIVE SUMMARY

Performance requirements for radioactive material (RAM) packages are specified in Title 10, Code of Federal Regulations, Part 71 (10CFR71). Package components forming containment boundaries must function in both the high- and low-temperature environments that are characteristic of a hypothetical fire accident and -40°F (-40°C) normal transport condition, respectively. Seals that provide the containment system interface between the packaging body and closure(s) are therefore a source of special consideration when designing and licensing a RAM package. Sandia National Laboratories conducted a research program to examine the performance during these temperature extremes of elastomeric O-ring seal materials commonly used in RAM packages. Performance characteristics examined included leakage rate versus temperature, compression set, hardness, and tracer gas permeation.

Face seal configuration fixtures were tested under static conditions and leak rates were measured at specific fixture temperatures using mass spectrometer leak detectors (MSLDs). Testing followed the practices and recommendations of American National Standards Institute (ANSI) N14.5 for leak testing using the helium back-pressurizing method. Because scaling laws for leakage rates do not exist, measurements from this program were intended to be used in a qualitative rather than as a quantitative measure of performance.

Low-temperature testing was performed using fixtures with standard groove dimensions recommended by Parker Seal Company. The series tested 26 compounds from 7 manufacturers and 9 parent chemical groups. Fixtures were cooled and leak-tested in 10°F (5.5°C) increments. Testing continued until gross failure or to -90°F (-68°C), the lower limit of the test chamber. Fixtures were tested at extremely low temperatures (in excess of regulatory requirements) to compare results to manufacturers' ratings.

For high-temperature testing, an alternate test technique was developed. The helium tracer gas that is responsible for the high sensitivity of the MSLDs rapidly and thoroughly permeates elastomers. This permeation, which increases with temperature, can mask leakage, thus rendering test results unusable. To address this problem, a residual gas analyzer (RGA) was used in conjunction with a tracer gas mixture of helium and neon. Helium provided the primary high-sensitivity signal, while the neon provided controlled interpretive data.

During the development tests, seals often expanded and completely filled the grooves. (The coefficient of thermal expansion for elastomers can be as much as ten times that of stainless steel.) Expansion coefficients for candidate materials were identified and maximum expansions were calculated based on target temperatures. Fixture groove widths were then increased to allow a maximum groove fill of 95%. Test fixtures were fabricated with groove designs specific to the material families. Groove depths were not altered; nominal compression remained unchanged. An abbreviated low-temperature test series showed the new groove designs did not adversely affect low-temperature performance.

Manufacturers' high-temperature ratings are typically based on a 1000-hour life, which decreases as temperatures increase. Target temperatures for this testing series were selected from seal life estimates made by Parker Seal Company for 10-hour use. These values exceeded the published ratings by 50 to 90°F (28 to 50°C), depending on compound family. The intent was to select temperatures for which there were high probabilities for success rather than testing to failure.

Test sequence for the high-temperature test series consisted of an assembly leak test at room temperature, a test upon reaching target temperature, a test after holding at target temperature for 2 hours, another test after cooling to ambient, and a final test at -40°F (-40°C). The first test at the target temperature verified that the seal did not fail during the temperature transient. The 2-hour dwell period simulated an extended fire scenario. (This simulation was conservative; both analysis

and actual fire tests showed the peak temperature periods to be considerably less than 2 hours.) Ambient, temperature tests were performed after the high-temperature testing to show integrity after cooling. The -40°F (-40°C) tests were added to complete the data; this step is not part of a hypothetical accident sequence.

Conclusions/Results

Low-temperature testing showed the need for careful material selection to meet RAM packaging criteria. Very few materials passed testing at or near the manufacturers' low-temperature rating. These ratings are typically based on passage criteria significantly different from RAM packaging requirements and should therefore not be relied upon without careful examination. However, the majority of materials tested did meet the basic -40°F (-40°C) criteria. The exception to this was fluorocarbon (viton), which failed sufficient tests to be of major concern. An added concern for low-temperature seal design that was not evaluated here is relative flange movement due to vibration or shock. A very hard or brittle seal would be unlikely to maintain leak-tightness if interface surfaces moved.

High-temperature tests showed that seal life estimates at elevated temperatures can be used to increase the upper limit of elastomeric materials. Every material tested passed leak tests at temperatures that were 50 to 90°F (28 to 50°C) higher than manufacturers' standard ratings. While reasonableness must be maintained when applying this philosophy to cask design, this range of increase in usable temperature can greatly aid in designing for an accident scenario.

Additional tests at 3-hour life ratings (an additional 30°F [17°C]) were performed. These tests were not intended to establish a precise upper limit of survivability for the seals, but rather to provide a level of confidence to the previous, 10-hour life testing. Results of these tests were also favorable.

Seals performed well in the groove designs used in this test series. It should be noted that groove designs from other seal manufacturers define slightly wider grooves, presumably with higher temperatures in mind. This test series showed that seal expansion at high temperature is an important consideration in seal groove design. To obtain satisfactory performance, maximum temperature and thermal coefficient of expansion should be calculated to ensure that the grooves are not overfilled.

1.0 INTRODUCTION

Radioactive material (RAM) packages must provide containment under a broad range of normal and hypothetical accident conditions. Test performance requirements are specified in Title 10, Code of Federal Regulations, Part 71 (10 CFR 71) (NRC, 1990). All regulatory conditions may be evaluated either by test or by analysis. Normal conditions include heat, cold, reduced and increased external pressure, vibration, water spray, free drop, compression, and penetration. Package performance at the maximum normal operating temperature is evaluated considering 100°F (38°C) ambient air temperature, solar insulation, and contents-generated heat. Low-temperature performance is evaluated considering a minimum ambient temperature of -40°F (-40°C) in still air in the shade. External pressures to be evaluated range from 24.5 kPa (3.5 psi) to 140 kPa (20 psi) absolute. Two other conditions that may affect package performance include vibration normally associated with transport and a free drop, usually from 1 ft (0.3 m). These conditions must not appreciably reduce the effectiveness of the package.

Evaluations of package performance in hypothetical accident conditions include free drop, puncture, thermal, and immersion tests. Acceptance of the package requires proven radiological performance for containment, shielding, and subcriticality. Package performance must be evaluated for a sequence of events consisting of a 30 ft (9 m) free drop onto an unyielding surface, a free drop of 40 in. (1 m) onto a puncture bar, and exposure to a flux equivalent to a thermal radiation environment of 1475°F (800°C) for a period of 30 minutes. A separate condition includes immersion of the package under 50 ft (15 m) of water for 8 hr. Determining the acceptability of RAM packages following the hypothetical accident sequence has been based on assuring that there is no leakage in excess of containment requirements, as demonstrated by leak testing.

The seals that provide the containment system interface between the packaging body and closure(s) are routinely a source of concern when designing, testing, and licensing a RAM package. Seals are usually elastomeric O-rings that can be considered delicate by cask component standards. These seals must not only perform under the rigorous conditions described above, but also are subject to multiple use through package opening and closing and human contact.

A seals technology program, jointly funded by the U.S. Department of Energy (DOE) Office of Defense Programs and the Office of Civilian Radioactive Waste Management (OCRWM), was initiated at Sandia National Laboratories (SNL) in late 1988. The program mission is to characterize the behavior and performance of seals commonly used in RAM packages under 10 CFR 71 (NRC, 1990) normal and hypothetical accident conditions. The objective of the test program is to establish a basic design guide for selecting suitable seal materials. A survey of cask designers revealed a need for data regarding seal performance under high-, low-, and cycled-temperature conditions. Other performance criteria include axial and horizontal movement of sealing surfaces, long-term compression set, and aging at operating temperatures. Specific seal materials (i.e., compounds) data were also gathered by the survey and incorporated into the test program. In addition, a compilation of seal materials used in existing casks (Warrant and Ottinger, 1989) was examined; while this information did not list specific compounds, particular compound families were common. Silicone was the predominant material, followed by neoprene, buna N (nitrile), and fluorocarbons.

A program was developed to test seal performance under high- and low-temperature extremes. Limited tests were also performed to evaluate compression set, permeation, and hardness. Due to limited time, personnel, and budget, no work was performed on simulated closure movement or aging.

This report documents the results of low-temperature testing performed in FY89 through FY92 and of high-temperature testing conducted in FY94. All tests were performed on face seal configuration fixtures under static conditions. In the face seal configuration, the compressive force is applied across the O-ring thickness, perpendicular to the inside diameter. Leak rates were measured at specific fixture temperatures using mass spectrometers and technical-grade tracer gases to compare seal material performances. Although leakage rate scaling laws have not been developed, it is probable that a compound that performed well in the tests reported herein will perform well in a full-scale package, given similar environmental and installation conditions.

The information in this report is presented in the following order: (1) a general description of testing, hardware, and equipment; (2) low-temperature testing; (3) high-temperature testing; (4) design guidance; and (5) a summary of the results and conclusions.

All testing was performed under SNL organizational and program-specific Quality Assurance (QA) Plans. All testing tasks were assigned and performed under a QA level 3, Minor. Testing adhered to numerous practices and requirements of higher Q-levels, including written and approved procedures, log book maintenance, and extensive use of National Institute of Standards and Technology (NIST)-traceable calibrated test and measurement equipment.

2.0 SEAL PERFORMANCE TEST DESCRIPTION

2.1 Seal Test Fixtures

The face seal design test fixture configuration is illustrated in Figure 1. Fabricated from 304 stainless steel, fixtures consist of a bottom plate, which contains two concentric O-ring grooves, and a flat top plate. These plates are bolted together with 24 0.25-in. (0.635 cm) capscrews. (Descriptions of O-ring groove designs and dimensions are detailed in Sections 3.1 and 4.2 for the low- and high-temperature test series.) Each fixture assembly provides for the installation of two 0.06-in. (0.152 cm)-diameter type K thermocouples to record fixture temperatures. Fixture fabrication drawings are contained in Appendix A.

2.2 Test Configuration

Figure 2 is a simplified schematic of the test configuration. For the low-temperature tests, a leak detector was piped to the cavity between the two seals and the tracer gas evacuation pump and supply were piped to the central cavity of the fixture. Before performing a test, the test cavity was monitored to assure stable background reading. The tracer gas cavity was evacuated and then backfilled with tracer gas. The detector was then monitored for leakage. This procedure follows the practices and recommendations of the American National Standards Institute (ANSI) for leak testing using the helium backpressurizing method (ANSI N14.5, 1987). Leak rates are reported in std cm^3/s (standard cubic centimeters per second); standard conditions are 32°F (0°C) and 14.7 psia (103 kPa).

Leakage monitored by the detector has two primary sources: (1) Bypass leakage, the gas flow across the seal/fixture interface, which is characterized by a response of the leak detector output signal starting within 2 s of the tracer gas injection. The signal then rises to an equilibrium value within a few, typically 10-20, seconds. The total rise time is controlled by the magnitude of the leak, the pumping speed of the detector, and the free volume of the piping. (2) Permeation leakage, the gas flow diffusing through the seal material, which is characterized by a delay in the response of the leak detector output signal ranging from 5 s to many minutes after tracer gas injection. The signal may take hours to reach an equilibrium value. Permeation time delay, rise time, and equilibrium rate are all dependent on the tracer gas, temperature, and seal material and size. See Section 4.1.

High-temperature tests also used this configuration, except the locations of the leak detector and tracer gas connections were switched. This switch reduces the background level of seal outgassing by decreasing the amount of seal surface area exposed to the detector and eliminates any possible permeation of tracer gas from the atmosphere.

Leakage rate measurements for the low-temperature tests were made with an Alcatel Model ASM-51 mass spectrometer leak detector (MSLD). This MSLD could have been used for the high-temperature tests, but since certain seal compounds demonstrate rapid helium permeation, it might have been difficult to distinguish between bypass and permeation leakage. As discussed in Section 4.1, a dual tracer gas mixture was selected for the high-temperature tests. The MSLD is not sensitive to the second tracer gas, neon, so it was necessary to use an Ametek Dycor residual gas analyzer (RGA) as the leak detector on the high-temperature tests. The output signals from both detectors were recorded by a computer-controlled data acquisition system.

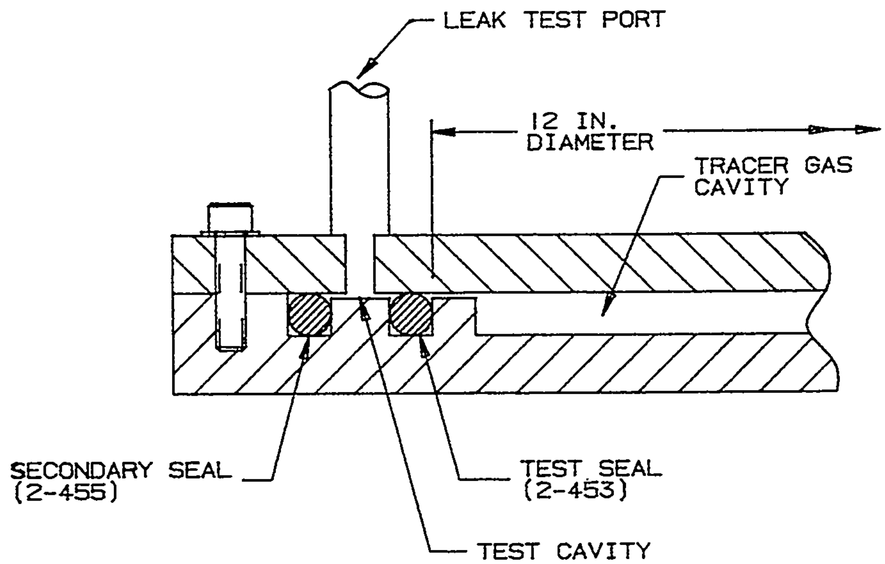

Figure 1. Seal Test Fixture

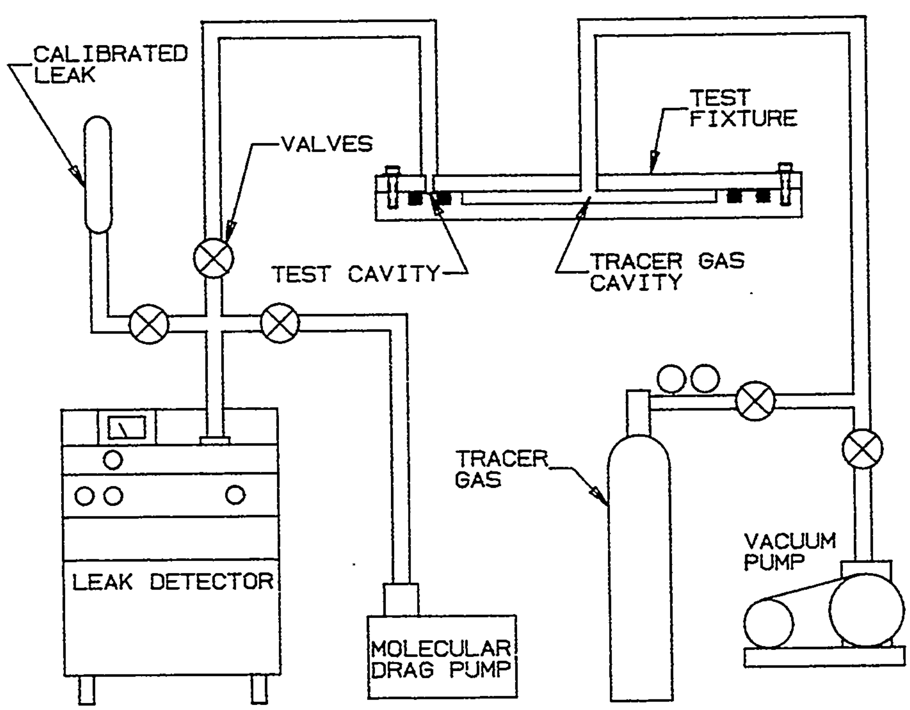

Figure 2. Leak Test Schematic

The leak test system, which allows placement of up to three fixtures in the environmental chamber, is piped to an extensive manifold system. This system, shown in Figure 3, uses electrically operated valves to control all leak test operations and the data acquisition system to log results.

2.3 Data Acquisition System

Two different data acquisition systems were used in the test program. The early (FY89-FY92) low-temperature testing used a Hewlett Packard (HP) Series 9000, Model 340 computer in conjunction with an HP 3852A data acquisition unit with software written specifically for this system. The software allowed the operator to maintain databases, control the remote valves, and record leakage rate and thermal data. Database information consisted of a test description; seal material type, size, and cure date; fixture utilization; calibration information; and thermocouple identification. A note pad subroutine was also available to the operator for recording actions and comments.

The later (FY94) high-temperature tests use an HP Series 9000, Model 360 with enhanced computational capabilities with the HP 3852A. New software was also developed to allow use of either the Alcatel MSLD or Dycor RGA. Other improvements include more user-friendly controls, the addition of a tracer gas pressure monitor, and extensive note and history logs. The history log automatically records real time data for every valve manipulation as well as other important system information. The pressure-monitoring routine automatically records tracer gas cavity evacuation and backfill pressures.

2.4 Test Hardware

In addition to the seal test fixtures, leak detectors, and data acquisition systems, several other pieces of equipment were used. Fixtures were thermally conditioned using two environmental chambers. A 6 ft^3 (0.17 m^3) Tenney Six chamber with an operating range of -150° to 390°F (-100° to 200°C) was used for all low-temperature testing. A Bemco chamber with 7 ft^3 (0.2 m^3) of volume and an operating range of ambient to 950°F (510°C) was used for the high-temperature tests.

Several devices were used to measure physical properties of the seals both pre- and post-test. Dimensional measurements were performed using a circumference-measuring fixture (Figure 4 and Appendix B), micrometers, and displacement transducers. A Shore Durometer, Model 714, with an operating range of 0 to 100 points with the A penetrator, was used for hardness measurements.

2.5 Measurement Uncertainty

Thickness measurements made on O-rings with machinist's micrometers have an uncertainty estimated as 0.002 in. (0.0051 cm) because of the difficulty of determining contact. Thickness measurements made with displacement transducers have an uncertainty of about 0.001 in. (0.0025 cm). Inside-diameter calculations have an estimated uncertainty of 0.015 in. (0.0381 cm) because of the frictional effects of the circumference-measuring device. Hardness measurement uncertainty is assumed to be 2 points.

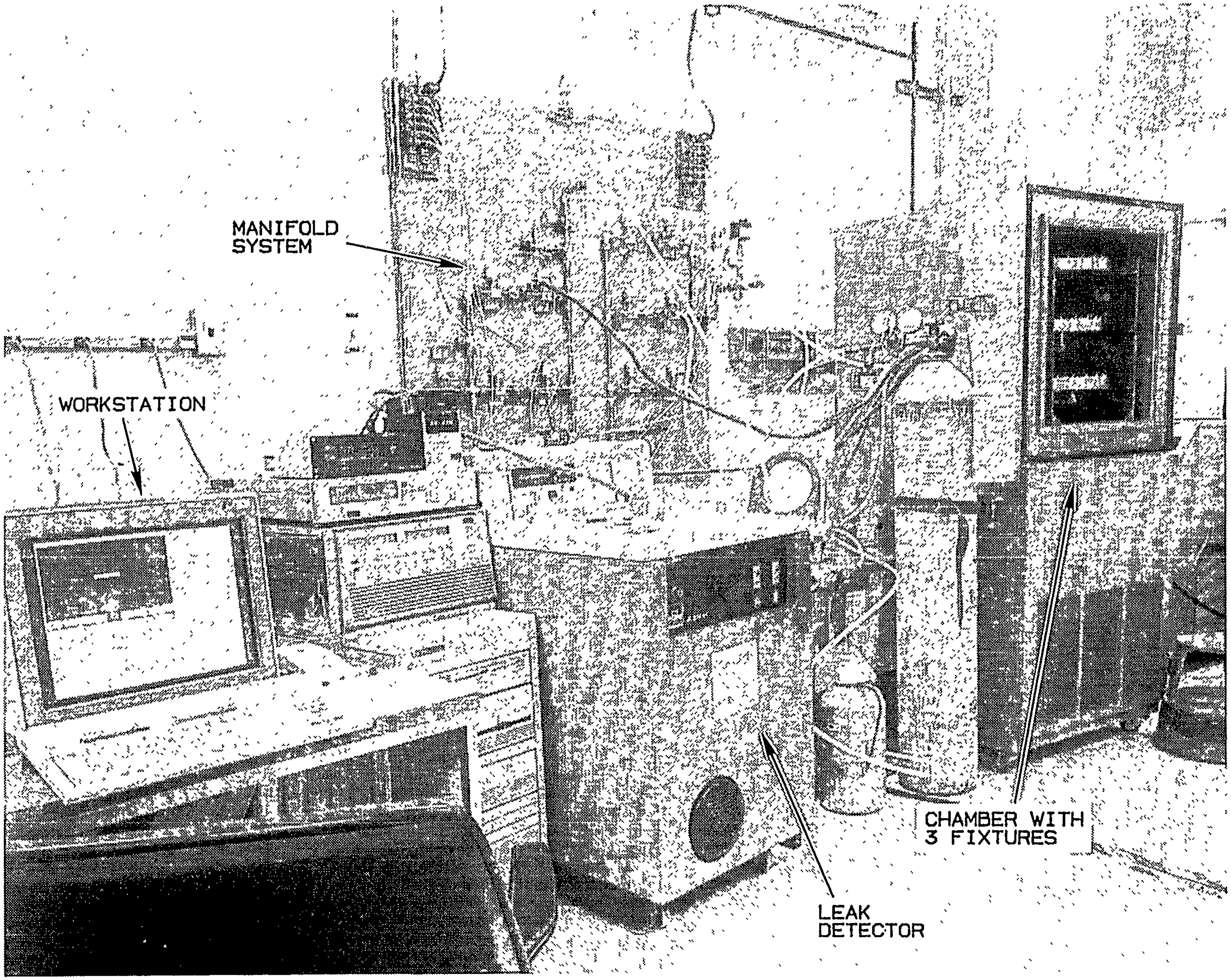

Figure 3. Seals Laboratory Leak Test System

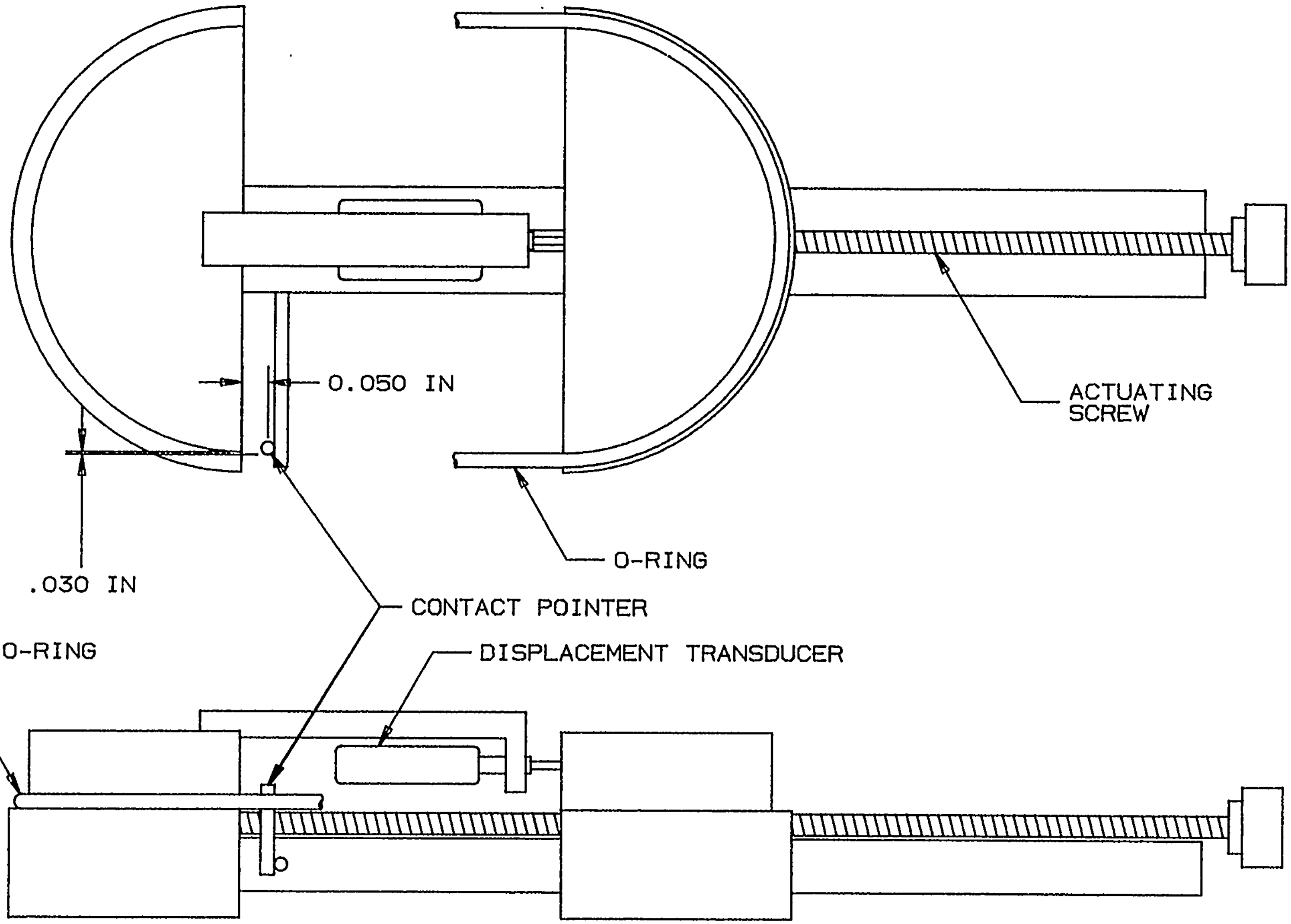

Figure 4. Seal Circumferential-Measurement Fixture

The SNL Primary Standards Laboratory assigns a 20% uncertainty to the calibration of standard leaks. This uncertainty combined with noisy signals, tracer gas partial pressure effects, and pumping speed variations leads to a total uncertainty estimate of 50% for leakage measurements. Because of the high uncertainty, leak rates are generally given in this report by one significant figure. In most cases, the leakage did not exceed the background reading; thus the background rate is given for those cases.

3.0 LOW-TEMPERATURE SEAL PERFORMANCE TESTS

Ambient-temperature permeation tests were performed on each material. Also for comparison, pre- and post-test dimensional measurements were taken of seal cross-sectional thicknesses and overall diameters. Low-temperature testing was then conducted and results were analyzed statistically. The Alcatel ASM-51 leak detector used for all tests had an operating range of about 5E-11 to 1E-5 std cm^3/s, with loss of operating pressure occurring at approximately 1E-4 std cm^3/s (gross leak).

3.1 Fixture Description

Nominal groove dimensions (Parker Seal Group, 1991) were used for the 2-453 (unified size number) inner (test) seal and the 2-455 outer (secondary) seal for the original set of low-temperature tests. Nominal seal dimensions are 0.273 in. (0.693 cm) thick by 12.0 in. (30.5 cm) or 13.0 in. (33 cm) inside diameter. The groove depth of 0.205/0.207 in. (0.521/0.526 cm) provided a nominal 25% compression, as specified by most manufacturers for vacuum/gas service. The surface finish was 16 microinches (0.4 μm) in the grooves and 64 microinches (1.6 μm) for the top plate seal surface. Undercuts were made on the bottom plate between the two grooves to allow tracer gas to pass across the void and around the fixture.

3.2 Seal Materials

Material selection for the low-temperature tests was based on a manufacturers literature review and a survey of cask designers. The wide variety of selected materials includes compounds from seven manufacturers and nine parent chemical groups or families. Composite materials included Teflon/silicone, a silicone core encapsulated with Teflon; Teflon/Viton, Teflon material around a Viton core; and Astro/Teflon, which uses a stainless steel spiral spring as the core. The 26 compounds tested and their manufacturers are listed in Table 1 by chemical family.

On Table 1, the two-digit dash number following the base compound number refers to material hardness (Shore A scale). The alpha designation groups the compounds by family, although each manufacturer has its own convention for this coding. Composite materials do not follow these conventions but rather are identified by name.

Seals used for the low-temperature tests (and later high-temperature tests) were purchased as off-the-shelf items during the 1988-1991 interval. Seals were stored in the laboratory under ambient conditions before and after tests.

3.3 Dimensional Measurements

O-ring cross-sectional thicknesses were measured using a small hand-held micrometer. Measurements made at four locations were averaged and recorded. Inside diameters were calculated using the fixture illustrated in Figure 4. To operate the seal circumferential-measurement fixture, an O-ring is placed over the fixture, and the fixture is then expanded while a small micrometer measuring the cross-sectional thickness of the seal at midspan is monitored. When a decrease in cross-sectional thickness is noted, the gap between the two fixture halves is measured with a caliper. At this point, the O-ring inner diameter can be calculated using a simple formula and recorded.

Table 1. Candidate Materials Selected for Low-Temperature Seal Performance Tests

Butyl		Polyphosphazene	
B0612-70	Parker	F0953-70	Parker
R0403-50	Rainier	R1801-70	Rainier
R0404-70	Rainier		
Ethylene Propylene		**Fluorocarbon**	
E0540-80	Parker	V747-75	Parker
E0740-75	Parker	V835-75	Parker
		R1429-70	Parker
		19657-GLT	Wynn's
		KALREZ 4079	duPont
Fluorosilicone		**Neoprene**	
L0677-70	Parker	C0873-70	Parker
		C1124-70	Parker
Silicone		**Teflon**	
S0383-70	Parker	NPTFE	Parker
S0604-70	Parker		
S0613-60	Parker		
S0899-50	Parker		
Composites			
Teflon[1]/Silicone	Chicago Gasket (CG)		
Teflon/Silicone	Creavey Seal (CS)		
Teflon/Silicone	Row		
Teflon/Viton[2]	CG		
Teflon/Viton	Row		
Astro[3]/Teflon	CS		

[1]Teflon is a trade name of duPont Company for tetrafluoroethylene.
[2]Viton is a trade name of duPont Company for fluorocarbon rubber.
[3]Astro is a trade name of Creavey Seal Company for their encapsulated wound stainless steel spring.

3.4 Permeation Tests

Helium permeation tests were performed on selected candidate seal materials to establish baseline data on initiation times. These data were used to estimate the time available for bypass leak measurements. Seals were assembled in the fixtures after pre-test dimensional measurements were completed. All tests were performed at the ambient temperature of 66° to 76°F (18° to 24°C). The test procedure was as described, except the helium tracer gas remained in the fixture for the duration of the test. Fixtures were monitored for initial permeation time (i.e., breakthrough). Stable saturation rates (equilibrium) and associated times (within specified time restraints) were also recorded.

3.5 Permeation Test Results

Figure 5 shows a typical permeation curve. The signal is flat (that is, below the detectable range) for a period of time. Initial breakthrough is indicated by the rapid signal rise. The permeation signal continues to rise and eventually levels off at an equilibrium saturation rate.

The test results for all selected materials are listed in Table 2. The first column lists the seal compound by family. The second column presents the permeation initiation or breakthrough time for each material. The third and fourth columns list the equilibrium saturation rate and the time required to reach equilibrium. All data are averaged by the number of seals tested (three or six).

Permeation initiation times varied from 2 min for silicone materials to no measurable permeation after 2 hr for C0873 neoprene material. While the data give comparative information on initiation times, the times for any given material are highly temperature-dependent.

Silicone saturation rates were significantly truncated when the rate quickly reached and exceeded the upper limit of the detector (1E-05 std cm^3/s). High saturation rates are not considered as much a problem as these data indicate, because the rates would greatly decrease with temperature reductions. Subsequent low-temperature tests showed that although high background rates from near-continual helium exposure were measured, test results were still usable. For example, silicone materials tested had low-temperature background rates due to saturation in the low E-07 std cm^3/s range.

3.6 Leak Test Procedure

Seals were cleaned with denatured alcohol, coated with a thin film of vacuum grease, and inserted into the fixture grooves. After bolting the fixtures together, they were placed in the low-temperature chamber and piped to the leak detector and tracer gas lines. Usually three fixture assemblies were placed in the chamber for simultaneous temperature conditioning.

Fixtures were cooled to an initial temperature of 20°F (-7°C), and individual fixture leak tests performed sequentially. Fixtures were then cooled in 10°F steps. A leak test was performed on each fixture at each step. Fixtures were tested to failure or to -90°F (-68°C), the lower limit of the chamber. Failure was defined as a gross leak (about 1E-4 std cm^3/s), large enough that the leak detector could not maintain a sufficient operating vacuum. Performance was tested at extreme low temperatures [in excess of the -40°F (-40°C) regulatory requirements] to obtain data for comparison to manufacturer usage ratings.

A copy of the formal test procedure is included in Appendix C.

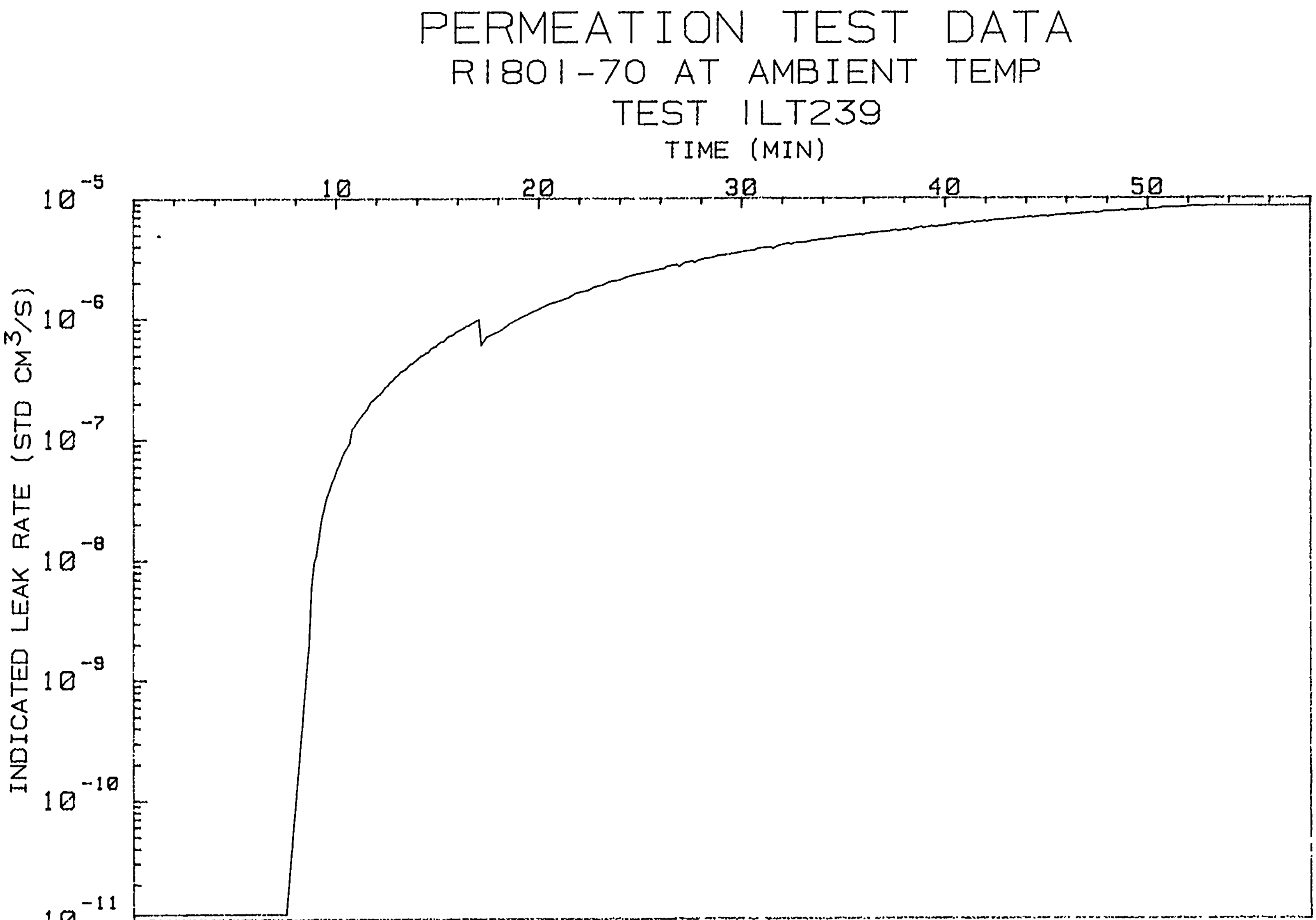

Figure 5. Example of Ambient-Temperature Permeation Data

Table 2. Ambient-Temperature Helium Permeation Data

Material	Average Permeation Initiation (min)	Permeation Equilibrium Rate (std cm^3/s)	Average Ambient-Temperature Equilibrium (min)
Butyl			
B0612-70	111	5E-07[1]	480
R0403-50	108	7E-09[1]	180
R0404-70	183	2E-08[1]	250
Ethylene Propylene			
E0540-80	74	5E-07	130
E0740-75	40	1E-06	120
Fluorocarbon			
V0747-75	71	3E-07	120
V0835-75	32	8E-07	150
R1429-70	21	8E-07	125
19657-GLT	37	7E-07	125
KALREZ 4079	14	6E-06	90
Fluorosilicone			
L0677-70	4	1E-05[2]	22
Neoprene			
C0873-70	>120[3]	NA[3]	NA[3]
C1124-70	41	4E-08[1]	120
Polyphosphazene			
F0953-70	6	1E-05[2]	60
R1801-70	9	1E-05[2]	53
Silicone			
S0383-70	3	1E-05[4]	10
S0604-70	3	1E-05[4]	10
S0613-60	2	1E-05[4]	12
S0899-50	2	1E-05[4]	8
Teflon			
NPTFE	49	2E-07[1]	120
Composites			
Teflon/Silicone-CG	6	7E-06	67
Teflon/Silicone-CS	12	9E-07	95
Teflon/Silicone-Row	7	9E-06	60
Teflon/Viton-CG	10	3E-6	105
Teflon/Viton-Row	14	1E-6	65
Astro/Teflon-CS	7	1E-06	115

[1]Equilibrium not reached due to time limitations.
[2]Equilibrium rate slightly abbreviated; detector range exceeded.
[3]No permeation detected.
[4]Equilibrium rate significantly abbreviated; detector range exceeded.

3.7 Leak Test Results

Figure 6 shows an example plot of a low-temperature leak test. In these tests, leakage rate data are plotted against temperature. This particular material had a minimal background rate of 1E-11 std cm^3/s at the 20°F (-7°C) initial test, and then a saturated background rate in the low E-07 std cm^3/s range until -85°F (-65°C), when failure (loss of vacuum) occurred.

A summary of low-temperature leak test data is presented in Table 3. The first column identifies the selected seal compounds separated by chemical families. The second column lists the total number of tests performed on each compound. Entries in the third column give the temperature ranges of failure. The fourth column list the average failure temperature for the material. The final column gives the manufacturer's advertised low-temperature rating for comparative purposes. This table contains data from all low-temperature tests performed and expands previously published early test results (Madsen et al., 1991).

With the exception of the silicone S0613 material, the seals were not consistently leaktight to the manufacturer low-temperature ratings, perhaps because most elastomeric seal applications are in the automotive, aircraft, or hydraulics industries. Most likely these industries have different performance criteria. Helium gas leakage tests are much more stringent and sensitive than typical tests for liquid leakage. For example, no visible water will leak from a known leak that passes dry air at a rate of 1E-04 std cm^3/s (ASNT, 1985).

Many compounds that did not perform to the manufacturer's ratings remained leaktight to the regulatory temperature requirement of -40°F (-40°C). Compounds that met the -40°F (-40°C) criterion were R0403, R0404, R1801, E0740, S0899, and Teflon/silicone materials by CG and by Row.

Several compounds, B0612, F0953, E0540, L0677, C1124, S0383, S0613, NPTFE, and both CS composites, had average failure temperatures at or below the -40°F target. In most cases, only 1 or 2 failures above -40°F (-40°C) kept them from routinely passing the target criterion.

Fluorocarbon (Viton) materials did not perform well, with very few individual tests passing the -40°F (-40°C) test step. Average failure temperatures were significantly higher. This was expected, as most of the compounds had only a -40°F (-40°C) rating.

A comprehensive statistical analysis performed on all low-temperature leak test data (see Appendix D) found that one of the test fixtures, B1, produced appreciably higher failure temperatures than the other fixtures. Later inspection showed a small nick in the sealing surface of the top plate. An estimated survival probability was established for each material after analyzing the data set with this fixture removed. This probability information was then used to select candidate materials for the high-temperature test series.

3.8 Dimensional Measurement Results

After testing, the fixtures were disassembled for inspection. Seals were allowed to relax for a minimum of 3 days before posttest measurements were performed. Dimensional data are presented in Table 4. The first column lists the seal compounds. The second and third columns list the average thickness change in inches and as a percentage of initial cross-sectional thickness (compression set). The fourth and fifth columns give the average change, in distance and percentage, for the overall seal diameter.

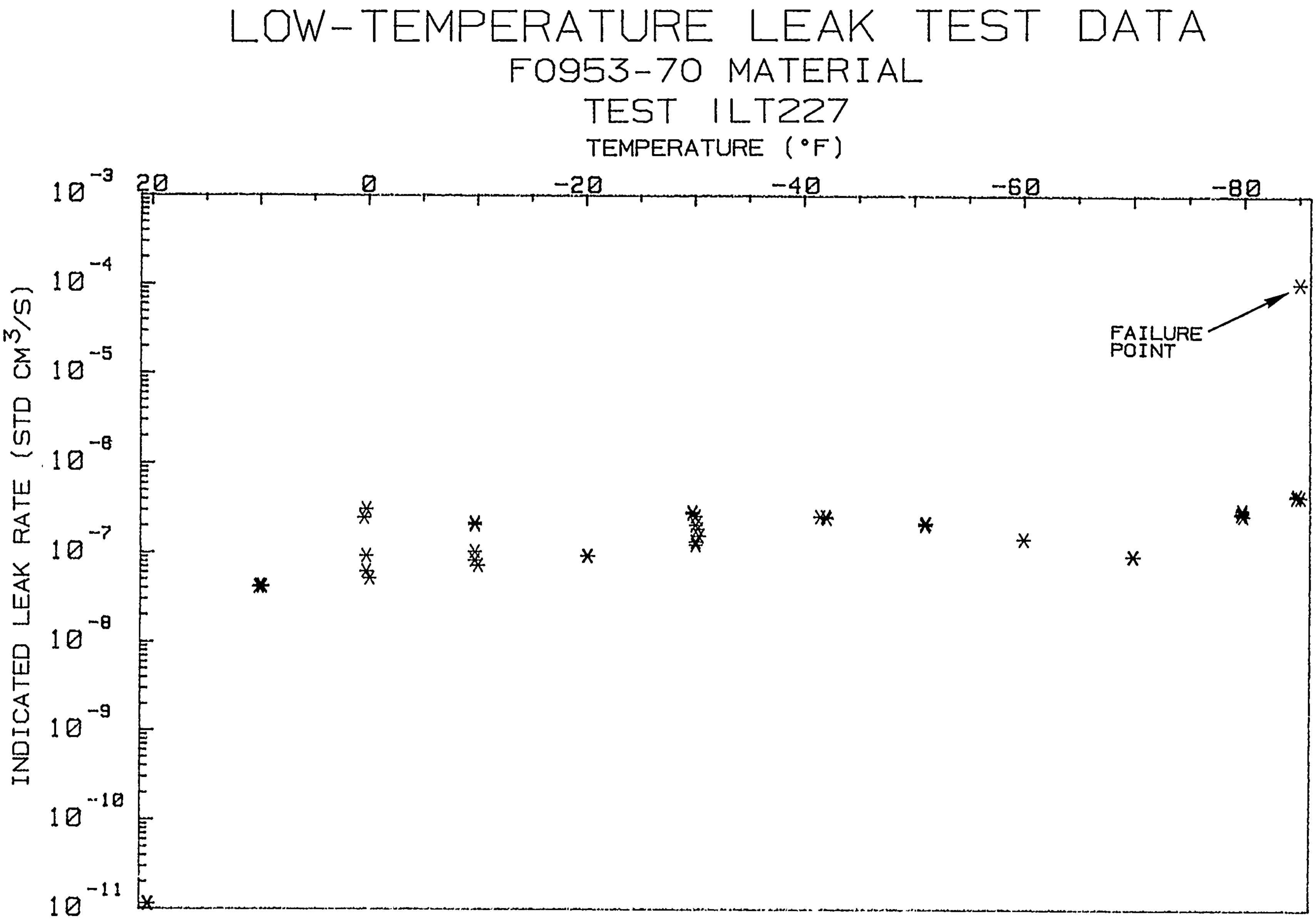

Figure 6. Example of Low-Temperature Leak Test Plot

Table 3. Low-Temperature Seal Performance Leak Test Data

Material	Number of Tests	Failure Temperature Range [°F (°C)]	Average Failure Temperature [°F (°C)]	Manufacturer Low-Temperature Rating [°F (°C)]
Butyl				
B0612-70	12	-10 to -83 (-23 to -64)	-68 (-56)	-75 (-60)
R0403-50	6	-50 to -68 (-46 to -56)	-63 (-53)	-65 (-54)
R0404-70	5	-40 to -68 (-40 to -56)	-53 (-47)	-65 (-54)
Ethylene Propylene				
E0540-80	9	-11 to -61 (-24 to -52)	-40 (-40)	-70 (-57)
E0740-75	6	-49 to -81 (-45 to -68)	-58 (-50)	-70 (-57)
Fluorocarbon				
V0747-75	6	+10 to -20 (-12 to -29)	-1 (-18)	-15 (-25)
V0835-75	17	+20 to -42 (-7 to -41)	-24 (-31)	-40 (-40)
R1429-70	6	+10 to -30 (-12 to -34)	-16 (-26)	-40 (-40)
19657-GLT	9	-19 to -31 (-28 to -35)	-27 (-33)	-40 (-40)
KALREZ 4079	5	+20 to +10 (-7 to -12)	+16 (-9)	-60 (-52)
Fluorosilicone				
L0677-70	9	-10 to -90 (-23 to -68)	-60 (-51)	-100 (-73)
Neoprene				
C0873-70	6	-30 to -41 (-34 to -41)	-34 (-37)	-45 (-43)
C1124-70	12	-36 to -71 (-38 to -57)	-51 (-46)	-65 (-54)
Polyphosphazene				
F0953-70	15	-1 to -85 (-18 to -65)	-60 (-51)	-85 (-65)
R1801-70	3	-60 to -80 (-51 to -62)	-73 (-58)	-85 (-65)
Silicone				
S0383-70	9	-1 to -90 (-18 to -68)	-46 (-43)	-100 (-73)
S0604-70	9	-1 to -65 (-18 to -54)	-35 (-37)	-65 (-54)
S0613-60	15	-70 to -71 (-56 to -57)	-70 (-56)	-60 (-51)
S0899-50	18	-41 to -92 (-41 to -69)	-85 (-65)	-100 (-73)
Teflon				
NPTFE	6	-17 to -90 (-27 to -68)	-52 (-47)	-40 (-40)
Composites				
Teflon/Silicone - CG	6	-49 to -65 (-45 to -54)	-54 (-48)	
Teflon/Silicone - CS	23	+20 to -90 (-7 to -68)	-45 (-43)	-80 (-62)
Teflon/Silicone - Row	6	-40 to -60 (-40 to -51)	-49 (-45)	-40 (-40)
Teflon/Viton - CG	9	+10 to -31 (-12 to -35)	-11 (-24)	
Teflon/Viton - Row	6	0 to -50 (-18 to -46)	-38 (-39)	-40 (-40)
Astro/Teflon - CS	6	-31 to -80 (-35 to -62)	-54 (-48)	-80 (-62)
Total Number of Tests	239			

Table 4. Low-Temperature Seal Performance Posttest Dimensional Measurements

Material	Average Compression Set [in.	(cm)]	Average Compression Set (%)	Average Diameter Change [in.	(cm)]	Average Diameter Change (%)
Butyl						
B0612-70	0.003	(0.007)	1.0	0.091	(0.231)	0.7
R0403-50	0.003	(0.007)	1.0	0.030	(0.076)	0.2
R0404-70	0.002	(0.005)	0.6	0.036	(0.091)	0.3
Ethylene Propylene						
E0540-80	0.003	(0.007)	1.0	0.025	(0.064)	0.2
E0740-75	0.002	(0.005)	0.6	0.000	(0.000)	0.0
Fluorocarbon						
V0747-75	0.006	(0.015)	2.2	0.018	(0.046)	0.2
V0835-75	0.002	(0.005)	0.9	0.034	(0.086)	0.3
R1429-70	0.004	(0.010)	1.3	0.020	(0.051)	0.2
19657-GLT	0.004	(0.010)	1.5	0.013	(0.033)	0.1
KALREZ 4079	0.003	(0.007)	0.9	0.058	(0.147)	0.5
Fluorosilicone						
L0677-70	0.003	(0.007)	1.3	0.188	(0.477)	1.6
Neoprene						
C0873-70	0.001	(0.003)	0.4	0.035	(0.088)	0.3
C1124-70	0.003	(0.007)	0.9	0.021	(0.053)	0.2
Polyphosphazene						
F0953-70	0.003	(0.007)	1.3	0.060	(0.154)	0.5
R1801-70	0.000	(0.000)	0.0	0.037	(0.094)	0.3
Silicone						
S0383-70	0.002	(0.005)	0.8	0.077	(0.196)	0.6
S0604-70	0.002	(0.005)	0.7	0.063	(0.160)	0.5
S0613-60	0.001	(0.003)	0.2	0.052	(0.132)	0.4
S0899-50	0.003	(0.007)	1.0	0.164	(0.417)	1.4
Teflon						
NPTFE	0.025	(0.063)	9.1	0.074	(0.188)	0.6
Composites						
Teflon/Silicone-CG	0.034	(0.086)	12.3	0.025	(0.064)	0.2
Teflon/Silicone-CS	0.011	(0.028)	3.9	0.028	(0.071)	0.2
Teflon/Silicone-Row	0.003	(0.007)	1.1	0.022	(0.056)	0.2
Teflon/Viton-CG	0.006	(0.015)	2.1	0.011	(0.028)	0.1
Teflon/Viton-Row	0.003	(0.007)	1.0	0.009	(0.023)	0.1
Astro/Teflon-CS	0.021	(0.053)	7.5	0.025	(0.064)	0.2

Compression sets for most seal compounds were low, varying from 0.001 in. (0.003 cm) to 0.004 in. (0.010 cm), less than 2%. This was true of the standard elastomers. Compression set for Teflon and Teflon composite materials varied widely, from 1% to 12%. These seals were much harder and had a significant set at disassembly. The variation in data is attributed to a very slow relaxation response that was influenced by the time between disassembly and inspection.

Diameter measurements of seals yielded data of little value. The measurements tended to have a large error margin, 0.015 in. (0.038 cm), compared to the simple thickness measurements, which were generally accurate to within 0.002 in. (0.005 cm). The largest diameter change of 0.188 in. (0.477 cm) was noted in L0677 material. These seals had the smallest pretest diameter and had to be stretched slightly for installation in the fixtures, which accounts for the change.

4.0 HIGH-TEMPERATURE SEAL PERFORMANCE TESTS

The high-temperature seal performance test series included four phases. First, several high-temperature scoping tests were performed to develop a reliable test technique using an RGA and a tracer gas other than helium. Information obtained from posttest observations showed that the seals were expanding and completely filling the grooves in many instances. This led to a redesign of test fixtures to widen the O-ring grooves. Next, an abbreviated low-temperature test series was conducted to ensure that the new groove design did not adversely affect low-temperature performance or test results. The actual high-temperature test series was then conducted, including pre- and post-test dimensional and hardness measurements. Last, an additional abbreviated high-temperature series was conducted at temperatures 30°F (17°C) higher than the main series.

4.1 <u>Test Procedure Development</u>

Preliminary scoping tests were performed on random materials at high temperatures to develop a reliable test technique. Helium permeation would be a severe problem at high temperature, conceivably masking bypass leakage rates. A test method was devised using an RGA in conjunction with an alternate (other than helium) tracer gas. Various gases (argon, neon, and sulfur hexafluoride) were tried before a mixture of helium and neon was selected. The equal partial-pressure mixture serves two functions. First, the helium signal provides the primary leak-rate measurement, because it has the highest signal-to-noise ratio and thus the highest sensitivity. Second, the neon 22 isotope can be monitored for response; a simultaneous rise in both signals indicates a bypass leak, while a delayed neon response denotes permeation (see Figure 7). This gas mixture is also unaffected by a liquid nitrogen cold trap required due to massive outgassing from most seal compounds.

A piping change was made to the test configuration as a result of the scoping tests. The leak detector was connected to the central cavity of the fixture, and the area between the two seals was used for the tracer gas cavity. This reduces the amount of seal surface area exposed to the detector, thus reducing outgassing rates. This configuration also eliminates the possibility of atmospheric neon permeating through the outer seal and raising test system background readings.

4.2 <u>Fixture Description</u>

Selected high-temperature tests used fixtures with modified groove designs. Early high-temperature scoping tests, performed primarily to evaluate detector capabilities and potential outgassing problems, yielded several seal failures. Upon fixture disassembly, certain seal compounds had expanded and completely filled the square-shaped groove; in some cases, the seal compound extruded from the groove to between the plates. Parker Seal Group (1991) warns that the coefficient of thermal expansion for some elastomers (primarily silicones) may be as much as ten times the coefficient for stainless steel. Other manufacturers avoid this problem by simply specifying wider groove widths, typically 0.345 to 0.380-in. (0.876 to 0.965 cm) for the 0.275-in. (0.699 cm) thickness O-rings (Wynn's Precision, Inc., 1993; Federal-Mogul Corp., 1988; and Apple Rubber Products, Inc., 1989). Thermal expansion coefficients for the candidate materials were identified and separated into three groups. Next, test temperatures were selected for the various candidate materials for which there was a high probability of success. Fixture groove widths were then increased to allow a maximum groove fill of 95%. This resulted in three

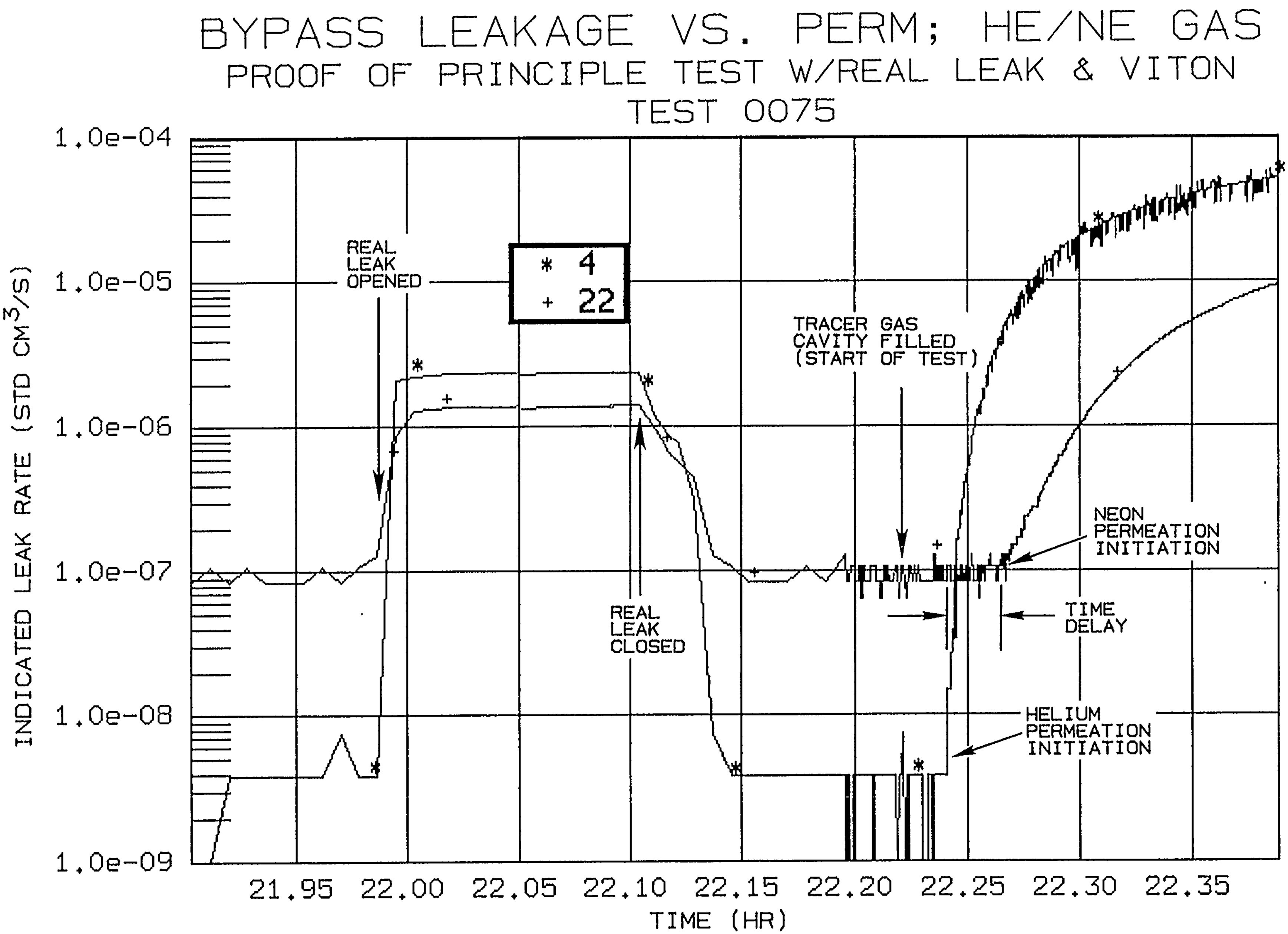

Figure 7. Example of Bypass Leakage Versus Permeation

different fixtures to be used for high-temperature tests: a standard/original groove width design for materials with low coefficient of thermal expansion and moderate maximum temperatures and 0.010 in. (0.025 cm) and 0.020 in. (0.051 cm) wider groove width fixtures for materials with higher coefficients and more extreme temperatures. Groove depths were not changed. The 21 fixture sealing surfaces were refaced during modification to remove damage found in the low-temperature series. Fabrication drawings of test fixtures can be found in Appendix A.

4.3 Seal Materials

Candidate material selections for the high-temperature tests were primarily based on results of the low-temperature series and the associated statistical analysis. The list of materials tested is shown in Table 5. The first column in this table lists the candidate materials. The third column gives the manufacturer-rated high temperature. The last column identifies the fixture configuration to be used for each specific compound, based on compound thermal coefficient of expansion (fourth column) and target temperature (second column).

The seals used for the high-temperature tests were generally new seals from the lots purchased for the low-temperature tests. These seals were manufactured between 1988 and 1991. In three cases, seals that were used in the low-temperature tests were reused in the high-temperature tests. Seals that were used in both tests are specified in the high-temperature seal performance test series matrix (Table 7).

In general, materials with a high probability of passing -40°F (-40°C) tests were included in this list. Added to the list of selected seal materials were V0835 (Viton) and E0893 (an ethylene propylene compound). Despite poor performance in the low-temperature testing, Viton is a popular material and additional information was desired. The ethylene propylene compound was added because this material had good temperature ratings, but was not available for the initial test series.

No composite materials were selected for this test series, due to the results of three Teflon/silicone material low-temperature tests performed using widened groove fixtures. Teflon compounds performed poorly in those tests. (Section 4.5 contains further information.)

Target temperatures were selected from seal life estimates by Parker Seal Group (1991) for 10-hr use. These values exceeded the published high-temperature ratings by 50 to 90°F (10 to 32°C), depending on compound family, because published high-temperature ratings typically relate to 1000-hr life. Temperatures were selected for which there were high probabilities for success; the goal was not to test to failure. Testing in increasing temperature steps until failure would yield ambiguous data because seal life is both time-dependent and temperature-dependent.

4.4 Widened Groove Low-Temperature Seal Performance Test Procedure

Fixtures were assembled, matching a fixture design (standard, +.010, or +.020) to a specific compound, as listed in Table 5. Although vacuum grease was used to lubricate the seals for the low-temperature tests, none was used for the high-temperature tests. The intent of this testing is to observe seal material performance alone, and effects of the grease could not be separated. Tests were conducted using the low-temperature Tenney chamber, Alcatel leak detector, and pure helium tracer gas.

Table 5. Candidate Materials Selected for High-Temperature Seal Performance Tests

Material	Target Test Temperature [°F (°C)]	Manufacturer High-Temperature Rating [°F (°C)]	Coefficient of Expansion [in./in./°F]	Fixture Design/Groove Width [in. (cm)]
B0612-70	300 (149)	250 (121)	6.2E-05	std/.305/.310 (.775/.787)
R0403-50	300 (149)	250 (121)[1]	6.2E-05[1]	std/.305/.310 (.775/.787)
R0404-70	300 (149)	250 (121)[1]	6.2E-05[1]	std/.305/.310 (.775/.787)
E0540-80	380 (193)	300 (149)	8.9E-05	+.010/.315/.320 (.800/.813)
E0740-75	380 (193)	300 (149)	8.9E-05	+.010/.315/.320 (.800/.813)
E0893-80	380 (193)	300 (149)	8.9E-05	+.010/.315/.320 (.800/.813)
C0873-70	380 (193)	300 (149)	7.6E-05	+.010/.315/.320 (.800/.813)
C1124-70	380 (193)	300 (149)	7.6E-05	+.010/.315/.320 (.800/.813)
V0835-75	470 (243)	400 (204)	9.0E-05	+.020/.325/.330 (.826/.838)
L0677-70	470 (243)	400 (204)[1]	1.0E-04	+.020/.325/.330 (.826/.838)
F0953-70	470 (243)	350 (177)	9.0E-05[1]	+.020/.325/.330 (.826/.838)
R1801-70	470 (243)	350 (177)[1]	9.0E-05[1]	+.020/.325/.330 (.826/.838)
Teflon/Silicone-Row[2]	470 (243)	400 (204)	8 to 9E-05[3]	+.020/.325/.330 (.826/.838)
Teflon/Silicone-CG[2]	470 (243)	400 (204)	8 to 9E-05[3]	+.020/.325/.330 (.826/.838)
Astro/Teflon-CS[2]	470 (243)	400 (204)	8 to 9E-05[3]	+.020/.325/.330 (.826/.838)
S0383-70	520 (271)	430 (221)	1.0E-04	+.020/.325/.330 (.826/.838)
S0604-70	520 (271)	450 (232)	1.0E-04	+.020/.325/.330 (.826/.838)
S0613-60	520 (271)	450 (232)	1.0E-04	+.020/.325/.330 (.826/.838)
S0899-50	520 (271)	430 (221)	1.0E-04	+.020/.325/.330 (.826/.838)

Coefficient of thermal expansion for stainless steel = 9.6E-06 in./in./°F

[1]Value was estimated by compound family; specific data unavailable from manufacturer.
[2]Deleted from high-temperature test due to results from low-temperature testing in widened groove fixture.
[3]Manufacturer data unavailable: value based on pure Teflon material.

An ambient-temperature leak test was performed to verify fixture assembly. Next, fixtures were cooled to 0°F (-18°C) for the initial low-temperature step and a leak test was performed. Fixtures were then cooled in 10°F increments, leak testing at each step until either a failure occurred or -60°F (-50°C) was achieved. Fixtures were not tested to the low-temperature extremes as in the initial series; rather, performance in the -40°F (-40°C) range was targeted.

Only compounds identified for testing in modified fixtures were included. Thus butyl compounds were not retested in this series, because no fixture change was needed for these materials.

4.5 Widened Groove Low-Temperature Seal Performance Test Results

Test results of compounds tested in modified groove fixtures are shown in Table 6. The first column lists the seal material. The number of tests performed on a specific compound is given in the second column. The third column specifies the modified fixture design, and the last column gives the final test temperature for which the seal remained leaktight. Specific failure temperatures are not identified as they were in the initial low-temperature series.

With the exception of the Teflon composite materials, all compounds performed as expected based on data from the initial low-temperature series and the associated statistical analysis. The only standard elastomer that did not regularly pass the test series to -60°F (-50°C) was V0835, failing above -40°F (-40°C) once and between -40°F (-40°C) and -50°F (-46°C) twice. This correlates with expectations for failure in the -30°F (-34°C) to -40°F (-40°C) range.

The Teflon composite materials all failed the initial 0°F (-18°C) test. Two of these three materials also failed an initial assembly test and had to be disassembled and reassembled. Possible causes for the failures are aging during storage, widened groove design, and assembly without lubrication. These seals had hardnesses in the 95 to 100 Shore A durometer range versus 50 to 80 for the standard elastomeric compounds. For these reasons, Teflon composite seals were dropped from the high-temperature series.

4.6 Dimensional Measurements

For the high-temperature test series, cross-sectional measurements were made using a platform-mounted displacement transducer with digital readout. Measurements were taken at four locations and averaged. Inside-diameter measurements were also made using the previously mentioned fixture in conjunction with a pointer at a fixed location (see Figure 4). After the O-ring was positioned, the fixture was expanded until the seal made contact with the pointer. The fixture gap was then recorded from the displacement transducer and the diameter calculated and recorded. Pre- and post-test hardness measurements were made for the high-temperature series to observe changes.

4.7 Leak Test Procedure

Appendix E contains the formal test procedure. A summary is given below.

Test sequence for the high-temperature test series consisted of an assembly leak test at ambient temperature, a test upon reaching a preselected target temperature, a test after holding at the target temperature for two hr, another test after cooling to ambient temperature, and a final leak test at -40°F (-40°C). The first test at the target temperature was included to verify that the seal did not fail during the temperature transient. The two-hr dwell period is a conservative estimate; both analyses and actual fire tests show the period to be considerably less than two hr. Ambient post-high-temperature tests were performed to show integrity after cooling. The -40°F (-40°C) tests were added for research purposes, as this step is not normally part of a hypothetical accident sequence.

Table 6. Widened Groove Low-Temperature Seal Performance Test Results

Material	Number of Tests	Fixture Design	Last Leaktight Temperature [°F (°C)]		
E0540-80	2	+.010	-60 (-50),	-60 (-50)	
E0740-75	2	+.010	-60 (-50),	-60 (-50)	
E0893-80	1	+.010	-60 (-50)		
C0873-70	2	+.010	-50 (-46),	-60 (-50)	
C1124-70	2	+.010	-60 (-50),	-60 (-50)	
V0835-75	3	+.020	-40 (-40),	-40 (-40),	-30 (-34)
L0677-70	3	+.020	-60 (-50),	-60 (-50),	-60 (-50)
F0953-70	3	+.020	-60 (-50),	-60 (-50),	-60 (-50)
R1801-70	2	+.020	-60 (-50),	-60 (-50)	
Teflon/Silicone/Row	1	+.020	0 (-18)		
Teflon/Silicone/CG	1	+.020	0 (-18)		
Astro/Teflon	1	+.020	0 (-18)		
S0383-70	2	+.020	-50[1] (-46),	-60 (-50)	
S0604-70	2	+.020	-50[1] (-46),	-50 (-46)	
S0613-60	2	+.020	-50[1] (-46),	-60 (-50)	
S0899-50	3	+.020	-50[1] (-46),	-60 (-50),	-60 (-50)
Total Number of Tests	32				

[1]Tested to -50°F only; does not indicate failure at -60°F.

Testing followed written procedures. Detector calibrations were performed prior to each test step using NIST-traceable standard leaks. Tracer gas was left in the test fixtures after the two-hr dwell at target temperature test to allow permeation for three purposes: First, at early time, it serves as the standard leak test; second, it demonstrates that the system is detecting and measuring the tracer gas mixture; and third, it provides permeation initiation data for both helium and neon at high temperatures.

Multiple batches (manufacturing lots) of a compound were included in the final test matrix (see Table 7) if such duplicates were available. The intent was to identify batch-to-batch variations, if possible with the limited quantities available. The matrix was also designed to allow posttest evaluation of fixture-caused effects by testing each compound (and each batch if duplicates were available) one time in each of three different fixtures.

Table 7. High-Temperature Seal Performance Test Series Matrix

Material	Batch	Test Number/Fixture			Test Temperature [°F (°C)]	Fixture Design
B0612-70	0804083	1/C1	2/C2	3/C3	300 (149)	Std
R0403-50[1]	49290	1/C2	2/C3	3/C1	300 (149)	Std
R0404-70[1]	52570	1/C3	2/C1	3/C2	300 (149)	Std
C1124-70	0809701	4/B1	7/B2	10/B3	380 (193)	+.010
E0540-80	0801225	4/B2	7/B3	10/B1	380 (193)	+.010
E0740-75	0803409	4/B3	7/B1	10/B2	380 (193)	+.010
E0740-75	0810101	5/B1	8/B2	11/B3	380 (193)	+.010
C0873-70	0810949	5/B2	8/B3	11/B1	380 (193)	+.010
C1124-70	0285844	5/B3	8/B1	11/B2	380 (193)	+.010
C0873-70	0800905	6/B1	9/B2	12/B3	380 (193)	+.010
E0893-80	0283995	6/B2	9/B3	12/B1	380 (193)	+.010
E0893-80	081976	6/B3	9/B1	12/B2	380 (193)	+.010
V0835-75	289464	13/A1	16/A2	19/A3	470 (243)	+.020
F0953-70	292539	13/A2	16/A3	19/A1	470 (243)	+.020
L0677-70	70762	13/A3	16/A1	19/A2	470 (243)	+.020
F0953-70	294683	14/A1	17/A2	20/A3	470 (243)	+.020
L0677-70	69700	14/A2	17/A3	20/A1	470 (243)	+.020
V0835-75[1]	291944	14/A3	17/A1	20/A2	470 (243)	+.020
L0677-70	70719	15/A1	18/A2	21/A3	470 (243)	+.020
V0835-70	291440	15/A2	18/A3	21/A1	470 (243)	+.020
R1801-70	17132	15/A3	18/A1	21/A2	470 (243)	+.020
S0383-70	68069	22/A1	25/A2	28/A3	520 (271)	+.020
S0899-50	69372	22/A2	25/A3	28/A1	520 (271)	+.020
S0613-60	69679	22/A3	25/A1	28/A2	520 (271)	+.020
S0613-60	70553	23/A1	26/A2	29/A3	520 (271)	+.020
S0604-70	69382	23/A2	26/A3	29/A1	520 (271)	+.020
S0899-50	69799	23/A3	26/A1	29/A2	520 (271)	+.020
S0899-50	70663	24/A1	27/A2	30/A3	520 (271)	+.020
S0383-70	70423	24/A2	27/A3	30/A1	520 (271)	+.020
S0604-70	70810	24/A3	27/A1	30/A2	520 (271)	+.020

Summary

B0612: 1 batch, 3 seals	V0835: 3 batches, 3 seals each
R0403: 1 batch, 3 seals	L0677: 3 batches, 3 seals each
R0404: 1 batch, 3 seals	F0953: 2 batches, 3 seals each
E0540: 1 batch, 3 seals	R1801: 1 batch, 3 seals
E0740: 2 batches, 3 seals each	S0383: 2 batches, 3 seals each
E0893: 2 batches, 3 seals each	S0899: 3 batches, 3 seals each
C0873: 2 batches, 3 seals each	S0613: 2 batches, 3 seals each
C1124: 2 batches, 3 seals each	S0604: 2 batches, 3 seals each

[1] These seals were previously used in a low-temperature test.

4.8 Leak Test Results

The vast majority of seals passed all tests by remaining leaktight, i.e., a leakage rate of less than 1.0E-07 std cm^3/s throughout the series. All test data from this series are presented in Table 8. Seal material is listed in the first column followed by the total number of tests performed on each material (batch lots included). The third through seventh columns present the average leakage rates at each of the five test steps. The final column lists the number of seal failures. Leak data were derived from the helium signal as described in Section 4.1. The neon 22 signal was used for interpretive and back-up purposes. Figure 8 shows a typical plot of high-temperature leak test data. Figure 9 illustrates a typical temperature plot.

Leakage rate values in Table 8 are background readings, not actual measured leakage rates, and are therefore defined as the lower limit of sensitivity. All values presented describe tests with no detectable leakage to that sensitivity.

Leakage rates/background values are averages of all tests of a given compound for which no leak was detected and the background rate is less than 1E-07. Individual tests with real (bypass) leakage indicated were omitted from the averages; instead, these tests were noted with test identification and circumstances specified. Any tests with background rates in excess of 1E-07 were also omitted from the averages and noted separately. The lower limit of sensitivity (helium) was typically in the low E-09 range for the RGA system. Seal outgassing and residual permeation from prior tests were the main contributors to higher backgrounds. Higher than normal background rates were evaluated by reviewing test logs and are explained in notes included in Table 8.

Most seals remained leaktight for all series tests with two significant exceptions. The F0953 and R1801 compounds failed 3 of 6 and 3 of 3 tests, respectively. These seals failed not at high temperature, but at the ambient or -40°F (-40°) tests. Both of these materials are polyphosphazene (trade name Eypel) compounds. High-temperature rating for this material was 350°F (177°C); the seals were overranged to 470°F (243°C). A dramatic decrease in material hardness and substantial compression set as a result of the tests was also noted. Additional test sets at lower temperatures were subsequently conducted with favorable results. Inquiries regarding the compound revealed that Ethyl Corporation, the licensee and maker of the base polymer, is not currently producing the material.

The single other seal failure in the test series was 1 seal of 9 tested of the S0899 silicone compound. This seal failed with a 6E-06 std cm^3/s rate for the -40°F (-40°C) test. Upon disassembly of the seal from the test fixture, a minor abrasion was noted. It is not known whether this damage was present during the test or caused at disassembly. This was the softest material in the test program, with a nominal hardness of 50, Shore A, and thus is easily abraded.

4.9 Dimensional Measurement Results

Dimensional measurement results are presented in Table 9. Seal material is identified in the first column. The second and third columns present cross-sectional thickness changes in inches and percentages. Inside-diameter change in inches and percentages are listed in the fourth and fifth columns. Values are averages of all seals tested.

Table 8. High-Temperature Seal Performance Leak Test Data

		Average Leakage Rates (std cm^3/s)					
Material	Number of Tests	Ambient/ Assembly	Initial at Temperature	Two-Hr at Temperature	Ambient/ Post-High-Temperature	-40°F (-40°C)	Number of Failures
B0612-70	3	<3E-09	<3E-08	<4E-08	<1E-08	<3E-09	0
R0403-50	3	<3E-09	<3E-08	<4E-08	<2E-08	<3E-09	0
R0404-70	3	<3E-09	<5E-09	<2E-08	<4E-09	<4E-09	0
E0540-80	6	<4E-09	<1E-08	<4E-08	<1E-08	<4E-09	0
E0740-75	6	<4E-09	<4E-09	<2E-08	<6E-09	<4E-09	0
E0893-80	3	<4E-09	<3E-08	<2E-08	<5E-09	<3E-09	0
C0873-70	6	<4E-09	<1E-08	<3E-08[1]	<7E-09	<4E-09	0
C1124-70	6	<4E-09	<5E-09	<5E-08	<5E-09	<4E-09	0
V0835-75	9	<5E-09	<8E-09	<1E-08	<1E-08	<2E-08	0
L0677-70	9	<4E-09[2]	<1E-08	<2E-08	<1E-08	<2E-08[3]	0
F0953-70	6	<5E-09	<8E-09	<1E-08	<1E-08[4]	<7E-09[5]	3
R1801-70	3	<2E-09[6]	<5E-09	<1E-08[7]	<2E-09[8]	ALL FAIL	3
S0383-70	6	<5E-09	<4E-09	<6E-09	<3E-09[9]	<2E-08	0
S0604-70	6	<4E-09	<4E-09	<6E-09	<4E-09	<7E-09	0
S0613-60	6	<4E-09[10]	<1E-08[10]	<9E-09[10]	<4E-09[10]	<6E-09[10]	0
S0899-50	9	<3E-09	<7E-09[11]	<6E-09[11]	<4E-09[11,12]	<6E-09[13,14]	1
Total Number of Tests	90						

[1]Test 8-B3/Two-hr dwell test: background of 1.5E-07 due to improper tracer gas evacuation after prior test (residual permeation).

[2]Test 16-A1/Assembly test: high initial background of 2E-07 due to insufficient cleanup time before starting test.

[3]Test 15-A1/-40°F test: 2E-07 background due to residual RGA contamination after a prior fixture failure (15-A3 at ambient).

[4]Test 20-A3/Ambient test: failed test; omitted from average.

[5]Tests 16-A3 and 19-A1/40°F test: failed test; omitted from average.

[6]Test 15-A3/Assembly test: 2E-07 background due to insufficient cleanup time before starting test.

[7]Test 15-A3/Two-hr dwell test: high 2E-07 background unexplained.

[8]Tests 15-A3 and 18-A1/Ambient test: both fixtures failed; value is for test 21-A2 only.

[9]Test 22-A1/Ambient test: very high background of 5E-06 due to permeation as result of an erroneous tracer gas backfill a short time earlier.

[10] Test 29-A3/All tests: atmospheric leak identified in test line after test; low to mid E-07 backgrounds; omitted from compound averages. Test sensitivity degraded to E-07 range.

[11]Test 22-A2/All tests except Assembly: atmospheric leak in test line gasket identified; led to low E-07 backgrounds.

[12]Test 22-A2/Ambient test: very high background of 4E-06 due to permeation as result of an erroneous tracer gas backfill a short time earlier.

[13]Test 22-A2/-40°F test: real bypass leak of low E-06 identified; omitted from average.

[14]Test 25-A3/-40°F test: 2E-07 background due to permeation; cross-plumbing tracer gas contamination from tests 25-A1 and 25-A2.

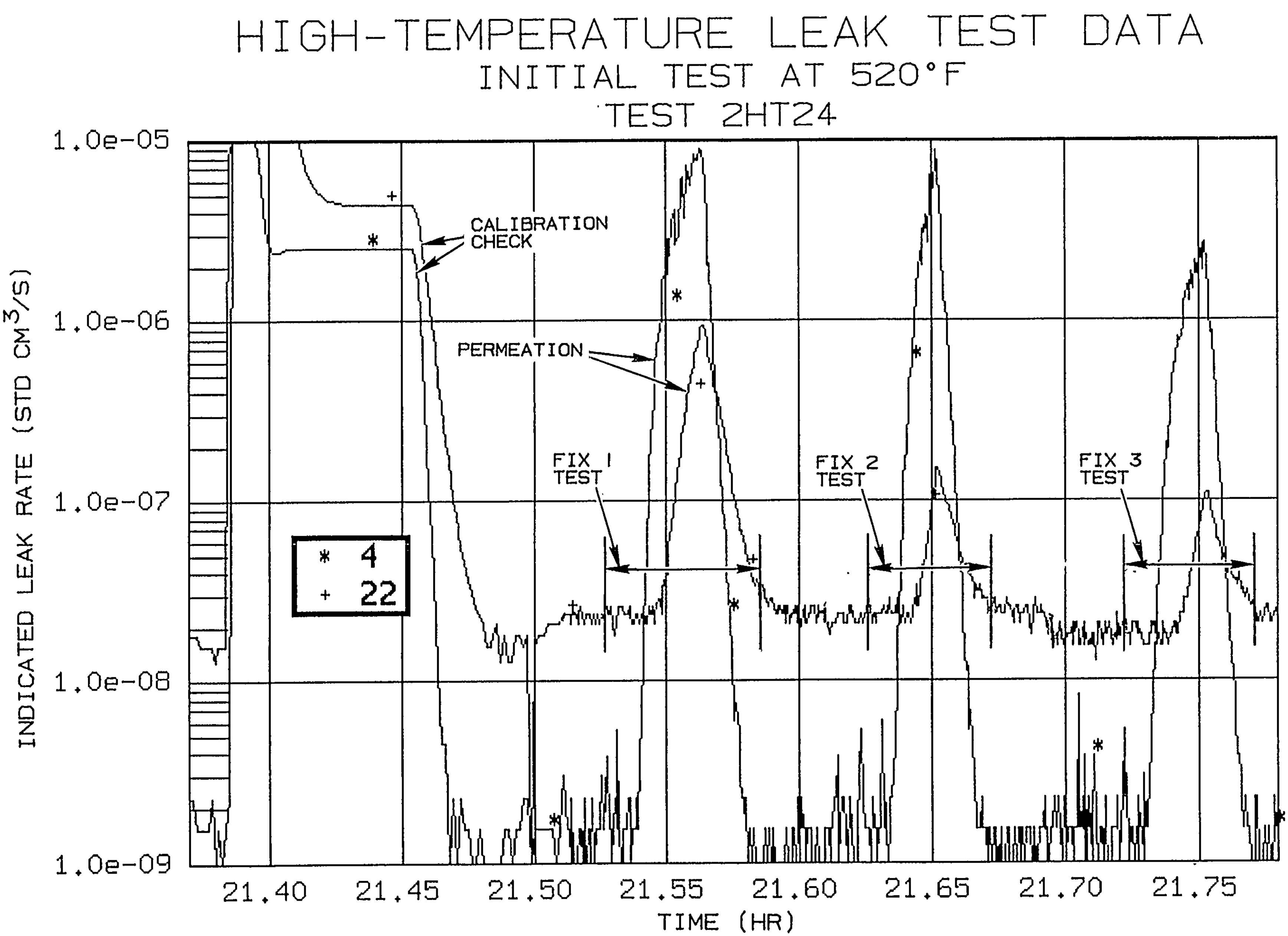

Figure 8. Example of High-Temperature Leak Test Data

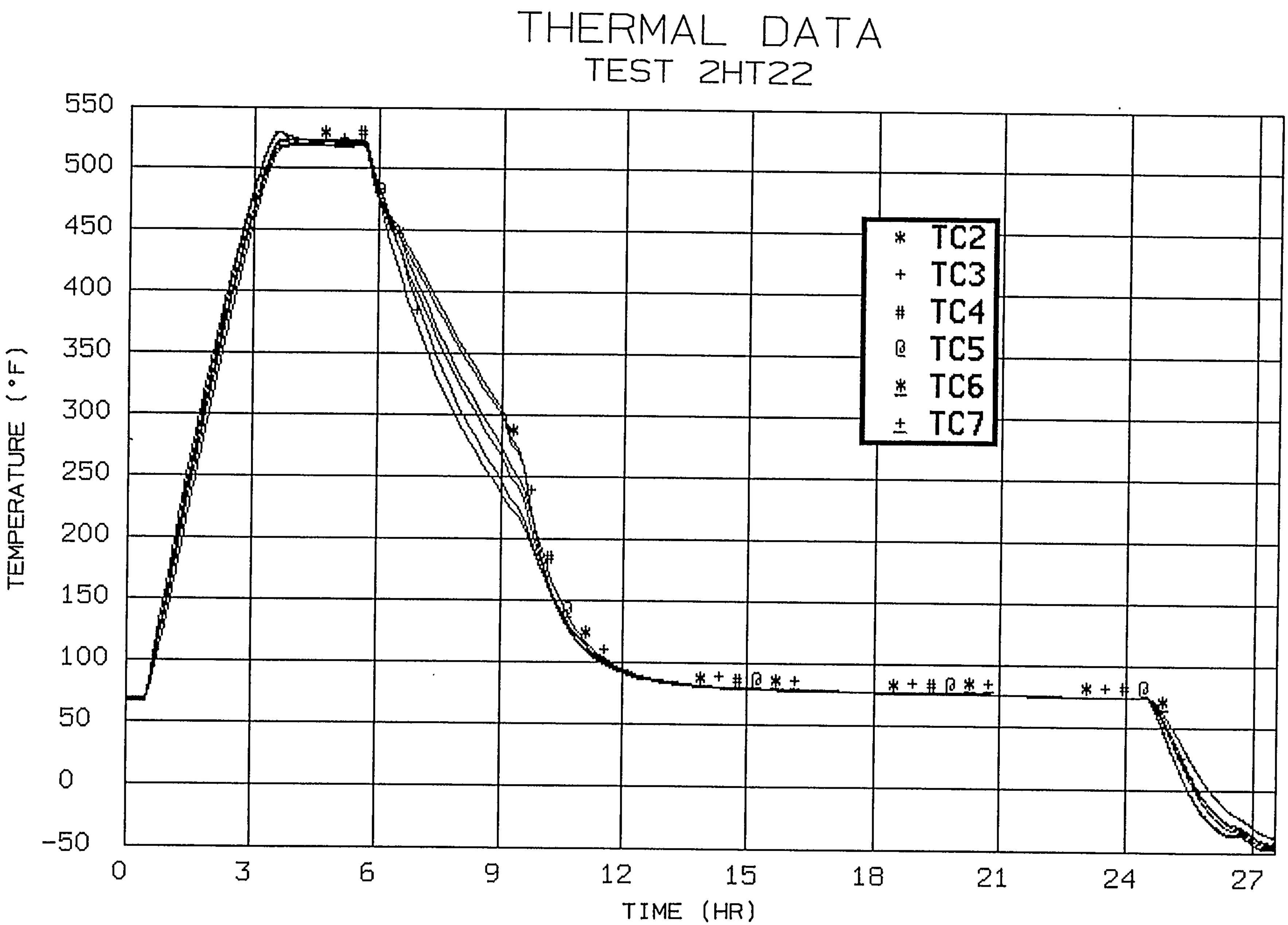

Figure 9. Example of High-Temperature Series Thermal Data

Table 9. High-Temperature Seal Performance Dimensional Data

Material	Average Compression Set [in. (cm)]	Average Compression Set (%)	Average Diameter Change [in. (cm)]	Average Diameter Change (%)
300°F (149°C) Tests/Standard Fixtures				
B0612-70	0.021 (0.053)	7.6	-0.03 (-0.08)	-0.2
R0403-50	0.021 (0.053)	8.2	-0.05 (-0.13)	-0.4
R0404-70	0.021 (0.053)	7.4	-0.08 (-0.20)	-0.7
380°F (193°C) Tests/+0.010 Fixtures				
E0540-80	0.035 (0.089)	12.8	-0.04 (-0.10)	-0.4
E0740-75	0.035 (0.089)	12.5	-0.07 (-0.18)	-0.5
E0893-80	0.020 (0.051)	7.2	-0.01 (-0.03)	-0.1
C0873-70	0.014 (0.036)	5.0	0.00 (0.00)	0.0
C1124-70	0.025 (0.064)	9.2	-0.01 (-0.03)	-0.1
470°F (243°C) Tests/+0.020 Fixtures				
V0835-75	0.017 (0.043)	6.3	0.01 (0.03)	0.1
L0677-70	0.016 (0.041)	5.8	0.03 (0.08)	0.3
F0953-70	0.053 (0.135)	19.6	-0.04 (-0.10)	-0.3
R1801-70	0.053 (0.135)	19.5	0.00 (0.00)	0.0
520°F (271°C) Tests/+0.020 Fixtures				
S0383-70	0.026 (0.066)	9.5	0.06 (0.15)	0.5
S0604-70	0.030 (0.076)	10.9	-0.04 (-0.10)	-0.3
S0613-60	0.022 (0.056)	8.2	-0.03 (-0.08)	-0.3
S0899-50	0.025 (0.064)	9.3	0.03 (0.08)	0.3

Compression set values varied from 0.014 in. (0.036 cm) (5.0%) to over 0.050 in. (0.127 cm) (nearly 20%), with all values generally following compound chemical families. The lowest compression sets of 0.014 to 0.025 in. (0.036 to 0.064 cm) (5% to 9%) were found in the butyl, neoprene, Viton, and fluorosilicone materials. Moderate compression sets of 8% to 11% were typical for silicone materials. Ethylene compounds (E0540, E0740, and E0893) were also moderate, at 9% to 13%. The largest changes took place in the polyphosphazene materials (F0953 and R1801), which had nearly 20% compressions sets. Polyphosphazenes are the only two materials that failed the high-temperature series.

Inside-diameter measurements were of little value. The seals remolded to fit the inside diameter of the groove. Those with larger diameters shrunk, while the smaller diameter seals enlarged.

Hardness test results are listed in Table 10. This table also presents data on helium and neon permeation initiation times. Data are averages of all tests on a particular compound.

Table 10. High-Temperature Seal Performance Hardness and Permeation Data

Material	Average Hardness Change (Shore A)	Permeation Initiation Time Helium (min)	Permeation Initiation Time Neon (min)
300°F (149°C) Tests/Standard Fixtures			
B0612-70	5.7	4.7	>7.0
R0403-50	3.7	5.5	>8.0
R0404-70	3.0	>8.0	>8.0
380°F (193°C) Tests/+0.010 Fixtures			
E0540-80	-1.3	1.8	4.6
E0740-75	0.7	1.7	4.0
E0893-80	-2.0	1.4	3.7
C0873-70	1.5	2.9	7.2
C1124-70	0.7	2.0	4.6
470°F (243°C) Tests/+0.020 Fixtures			
V0835-75	-0.1	1.1	2.9
L0677-70	-1.9	0.6	1.4
F0953-70	-18.3	1.0	2.4
R1801-70	-20.3	1.4	2.9
520°F (271°C) Tests/+0.020 Fixtures			
S0383-70	-2.0	0.5	1.1
S0604-70	-1.7	0.5	1.2
S0613-60	-2.2	0.6	1.4
S0899-50	-1.4	0.5	1.2

Most compounds showed little significant changes, with consistent results along chemical family lines. Hardnesses increased by about 4 points (Shore A) for the butyl compounds. Silicone hardnesses decreased by approximately 2 points for each compound. Ethylene, neoprene, fluorocarbon, and fluorosilicone had no significant changes. Such minor changes are favorable, because they suggest that significant chemical changes are not taking place. Chemical changes are not reversible (Madsen et al., 1991).

As with compression set, considerable changes were noted for the polyphosphazene compounds. These two materials softened by 18 to 20 points, with posttest hardnesses in the 50 to 60 range. While these final values are comparable to the softer silicone compounds, the seals were very pliable to the touch and appeared to have very little resilience or elasticity.

Permeation data are also presented in Table 10. These permeation initiation times are averages of measurements made at the two-hr dwell test step. Most data were not obtained for butyl compounds, because collecting data required holding the seals at temperature for a period significantly longer than the two-hr test requirements.

The RGA used to determine permeation times has a helium sensitivity about two orders of magnitudes less than that of the Alcatel ASM-51 (i.e., approximately E-9 versus E-11 std cm^3/s). Thus the times presented in Table 10 are determined at a permeation rate about 100 times larger than the rate for times presented in Table 2. The higher threshold determines the large difference.

Results were as expected; neon initiation times were a little over twice as long as for helium. This follows from classical kinetic gas theory, which shows that diffusion time varies directly with the square root of the molecular weight, other factors being equal. The square root of the molecular weight ratio is 2.2, reasonably consistent with the data.

The short permeation times for silicone illustrate a drawback in using helium as a tracer gas, especially at high temperatures. Theory also indicates that diffusion time is inverse with the square root of absolute temperature. If the RGA had the same helium sensitivity as the Alcatel ASM-51, the helium initiation times would have been only a few seconds.

4.10 High-Temperature Retesting

Three compounds were selected for retesting at revised temperatures. The two polyphosphazene compounds, which failed the original series, were selected based on a reevaluation of the manufacturer rating versus tested temperature. Fluorosilicone was also selected for retest at a higher temperature, based on test results that compared closely to the silicone compounds.

Adjusted test temperatures, fixture utilization, and the test matrix for retesting are shown in Table 11. The same test procedure and sequence were followed as the initial testing series, except that pre- and post-test diameters were not measured. The fixture was changed for the polyphosphazene compounds, which used +0.020-in. (0.051-cm) groove fixtures in the original series.

Table 11. High-Temperature Seal Performance Test Matrix - Retests at Adjusted Temperatures

Material	Batch	Test Number/Fixture			Test Temperature [°F (°C)]	Fixture Design
F0953-70	292539	31/B1	32/B2	33/B3	380 (193)	+.010
F0953-70	294683	31/B2	32/B3	33/B1	380 (193)	+.010
R1801-70	17132	31/B3	32/B1	33/B2	380 (193)	+.010
L0677-70	70762	34/A1	35/A2	36/A3	520 (271)	+.020
L0677-70	69700	34/A2	35/A3	36/A1	520(271)	+.020
L0677-70	70719	34/A3	35/A1	36/A2	520(271)	+.020

4.11 High-Temperature Retesting Results

Results of leak testing are presented in Table 12. Table 13 is a combined listing of dimensional, hardness, and permeation data. As in previous tests, data are averages of tests conducted on a particular compound. No abnormal backgrounds occurred.

All seals passed all leak tests with no detectable leakage above the prevailing backgrounds.

Polyphosphazene compounds performed well at the lowered temperatures. Compression set values were much improved, at less than 8% versus nearly 20% in prior testing. Material hardness change also improved, with a decrease of approximately 7 points versus 20 points earlier.

Table 12. High-Temperature Seal Performance Leak Test Data - Retests

		Average Leakage Rates (std cm^3/s)					
Material	Number of Tests	Ambient/ Assembly	Initial at Temperature	Two-Hr at Temperature	Ambient/ Post-High-Temperature	-40°F (-40°C)	Number of Failures
F0953-70	6	<3E-09	<4E-09	<6E-09	<3E-09	<1E-08	0
R1801-70	3	<3E-09	<5E-09	<3E-09	<4E-09	<1E-08	0
L0677-70	9	<3E-09	<3E-09	<6E-09	<4E-09	<6E-09	0
Total Number of Tests	18						

Table 13. High-Temperature Seal Performance Inspection and Permeation Data - Retests

Material	Average Compression Set [in. (cm)]	Average Compression Set (%)	Average Hardness Change (Shore A)	Permeation Initiation Time Helium (min)	Permeation Initiation Time Neon (min)
380°F (193°C) Tests					
F0953-70	0.020 (0.051)	7.5	–6.7	1.0	2.6
R1801-70	0.022 (0.056)	7.9	–7.7	1.4	3.9
520°F (271°C) Tests					
L0677-70	0.026 (0.066)	9.6	–7.0	0.5	1.3

The L0677 material compression set increased from 6% in prior tests to nearly 10%. Material hardness also changed significantly, with a softening of 7 points as compared to a decrease of 2 points earlier. These values suggest that while the seals passed all tests, the elevated test temperature might be near the upper limit of survivability.

Permeation data were little changed from previous tests. Initiation times were as expected, with the trends showing slight decreases with increased temperatures and increased times with lowered temperatures.

4.12 Additional High-Temperature Leak Tests

Another and final high-temperature test series was performed. This abbreviated series tested a single seal from each compound and batch at a temperature 30°F (17°C) higher than the last test for each material. These temperatures were chosen based on an approximate three-hr use estimated by Parker Seal Group (1991), in life versus temperature data.

These tests are not intended to establish a precise upper limit of survivability for the seals, but rather to provide a level of confidence to the previous testing. The underlying assumption of these tests is if seals pass these tests with regularity, it would indicate they were not on the verge of failure in the earlier tests.

Test sequence and procedure remained unchanged from prior high-temperature tests. Cross-sectional-thickness measurements were the only inspections performed. The test matrix shown in Table 14 identifies test temperatures and fixture utilization.

4.13 Additional High-Temperature Test Results

All seals with the exception of one fluorosilicone (L0677) performed well, passing all series tests. The one L0677 (of three tested) failed at the -40°F (-40°C) test step. The test temperature for this material had already been increased significantly above the manufacturer rating in the previous retests. Final test temperature was far in excess of the 1000-hr-life rating of 350°F (177°C) and of an estimated 460°F (238°C) 3-hr-life rating. Because the estimated 3-hr-life temperature of approximately 460°F (238°C) was also exceeded, failures were not unexpected.

Leak data are presented in Table 15, with the leak values given actually representing no detectable leakage to that sensitivity level. All data are averages.

Compression set and high-temperature permeation data are given in Table 16. The table lists material, compression set in inches, and compression set in percentage in the first three columns. The last column presents helium and neon permeation initiation time.

As a result of the increased temperature, butyl material compression set increased, to 10 to 11% from a previous 7 to 8%. Butyl is still believed to be in an acceptable range for probable survival. Viton (V0835) showed little change, with compression set increasing a fraction of a percent. Ethylene materials ("E" compounds) data were erratic. No explanation for these inconsistent results was found. Polyphosphazene materials also showed sharp increases, to approximately 14%. The comparative values of approximately 8% are from the retesting performed at 380°F (193°C). Compression sets were near 20% (versus design compression of 25%) after the original tests series at 470°F (243°C), where these seals failed.

C0873 and C1124 neoprene compound compression sets increased dramatically, nearing 17% in the final tests. This value may indicate that although the seals passed testing, the 410°F (210°C) test temperature may be excessive.

Silicone materials (S and fluorosilicone L) behaved consistently as a group. Compression sets increased to a 12 to 15% range from a previous 8 to 11%. Again, this range of compression set suggests that the final test temperature is near the upper limit of survivability. One L0677 failed.

Table 14. Added High-Temperature Seal Performance Test Series Matrix

Material	Batch	Test Number/ Fixture	Material	Batch	Test Number/ Fixture
330°F (160°C) Tests/Standard Fixtures			500°F (260°C) Tests/+0.020 Fixtures		
B0612-70	0804083	37/C1	V0835-75	0809819[1]	42/A1
R0403-50	49290	37/C2	V0835-75	291944	42/A2
R0404-70	52570	37/C3	V0835-70	291440	42/A3
410°F (210°C) Tests/+0.010 Fixtures			550°F (288°C) Tests/+0.020 Fixtures		
C1124-70	0809701	38/B1	S0383-70	68069	43/A1
E0540-80	0801225	38/B2	S0899-50	69372	43/A2
E0740-75	0803409	38/B3	S0613-60	69679	43/A3
E0740-75	0810101	39/B1	S0613-60	70553	23/A1
C0873-70	0810949	39/B2	S0604-70	69382	23/A2
C1124-70	0285844	39/B3	S0899-50	69799	23/A3
C0873-70	0800905	40/B1	S0899-50	70663	24/A1
E0893-80	0283995	40/B2	S0383-70	70423	24/A2
E0893-80	081976	40/B3	S0604-70	70810	24/A3
F0953-70	292539	41/B1	L0677-70	70762	46/A1
F0953-70	294683	41/B2	L0677-70	69700	46/A2
R1801-70	17132	41/B3	L0677-70	70719	46/A3

[1]Odd batch: does not match prior test matrix; only one seal available.

Table 15. High-Temperature Seal Performance Leak Test Data - Three-Hr Rating

Material	Number of Tests	Average Leakage Rates[1] (std cm^3/s) Ambient/ Assembly	Initial at Temperature	Two-Hr at Temperature	Ambient/ Post-High Temperature	-40°F (-40°C)	Number of Failures
330°F (166°C) Tests/Standard Fixtures							
B0612-70	1	<2E-09	<8E-08	<6E-08	<8E-09	<4E-09	0
R0403-50	1	<3E-09	<9E-09	<2E-08	<2E-08	<3E-09	0
R0404-70	1	<2E-09	<5E-09	<2E-08	<3E-09	<4E-09	0
410°F (210°C) Tests/+0.010 Fixtures							
E0540-80	2	<3E-09	<6E-09	<2E-08	<1E-08	<4E-09	0
E0740-75	2	<3E-09	<6E-09	<1E-08	<4E-09	<3E-09	0
E0893-80	1	<3E-09	<4E-09	<2E-08	<5E-09	<5E-09	0
C0873-70	2	<3E-09	<8E-09[2]	<5E-08	<3E-09	<3E-09	0
C1124-70	2	<2E-09	<1E-08	<5E-08	<3E-09	<3E-09	0
F0953-70	2	<3E-09	<8E-09	<9E-09	<3E-09	<2E-08	0
R1801-70	1	<2E-09	<1E-08	<4E-08	<3E-09	<2E-08	0
500°F (260°C) Tests/+0.020 Fixtures							
V0835-75	3	<3E-09	<3E-09	<3E-09	<3E-09	<3E-09	0
550°F (280°C) Tests/+0.020 Fixtures							
L0677-70	3	<3E-09	<6E-09	<4E-09	<4E-09	<3E-08[3]	1
S0383-70	2	<3E-09	<3E-09	<3E-09	<3E-09	<4E-09	0
S0604-70	2	<3E-09	<3E-09	<4E-09	<3E-09	<4E-09	0
S0613-60	2	<3E-09	<3E-09	<3E-09	<3E-09	<4E-09	0
S0899-50	3	<3E-09	<3E-09	<3E-09	<3E-09	<5E-09	0
Total Number of Tests	30						

[1] As applicable; some data are from a single test.
[2] Test 40-B1/Initial high-temperature test: background of 1E-07; cause unknown.
[3] Test 46-A2/-40°F test: failed.

Table 16. High-Temperature Seal Performance Inspection and Permeation Data - Three-Hr Rating

Material	Average[1] Compression Set [in.	(cm)]	Average[1] Compression Set	Permeation Initiation Time Helium (min)	Neon (min)
330°F (166°C) Tests/Standard Fixtures					
B0612-70	0.031	(0.079)	11.2	4.5	>6.0
R0403-50	0.026	(0.066)	10.1	>5.0	>5.0
R0404-70	0.028	(0.071)	10.0	>5.0	>5.0
410°F (210°C) Tests/+0.010 Fixtures					
E0540-80	0.033	(0.084)	11.7	1.6	3.8
E0740-75	0.011	(0.028)	3.8	1.3	3.3
E0893-80	0.033	(0.084)	12.0	1.4	3.0
C0873-70	0.044	(0.112)	15.9	2.7	>5.0
C1124-70	0.047	(0.119)	17.0	1.8	4.3
F0953-70	0.039	(0.099)	14.2	0.9	2.6
R1801-70	0.037	(0.094)	13.6	1.4	3.7
500°F (260°C) Tests/+0.020 Fixtures					
V0835-75	0.018	(0.046)	6.7	1.0	2.4
550°F (288°C) Tests/+0.020 Fixtures					
L0677-70	0.039	(0.099)	14.5	0.5	1.2
S0383-70	0.037	(0.094)	13.8	0.5	1.1
S0604-70	0.041	(0.104)	15.1	0.5	1.2
S0613-60	0.037	(0.094)	13.5	0.5	1.2
S0899-50	0.033	(0.084)	12.2	0.6	1.2

[1]As applicable; some data are from a single test.

5.0 DESIGN GUIDANCE

Based on the performance testing described in this report, the following guidance is offered for cask designers' consideration.

5.1 Groove Design

Basic rectangular groove designs with 25% compression, as recommended by the manufacturers, are satisfactory to meet the low- and ambient-temperature requirements. However, increasing compression from 25% to 30-35% may help the fluorocarbons to seal more effectively at low temperature.

The designer should check the volumetric groove fill ratio at the maximum expected temperature to verify that the ratio is in a suitable range, preferably no higher than 0.95. The following simplified equation can be used for the calculation:

$$\text{Fill Ratio} = \frac{\pi \times w^2 \times (1 + 3 \times \alpha_E \times \Delta T)}{4 \times H \times G \times (1 + 3 \times \alpha_M \times \Delta T)}$$

where

w = nominal O-ring cross section diameter,

H = distance from groove bottom to outer seal surface (groove depth for a face seal; groove depth + clearance for a radial seal),

G = groove width,

α_E = linear coefficient of thermal expansion for the elastomer,

α_M = linear coefficient of thermal expansion for the body material, and

ΔT = temperature difference from ambient (T_{max} - $T_{ambient}$).

Units must be consistent with those of the coefficients. The coefficients may be found in materials handbooks or in some of the O-ring manufacturers' catalogs.

Surface finishes and machining lays recommended by the manufacturers were satisfactory in these tests.

5.2 Compound Selection

Of the compound families tested, butyl, ethylene propylene, fluorocarbon, fluororsilicone, neoprene, polyphosphazene (no longer available as of May 1994, according to the Ethyl Corporation), silicone, Teflon, and Teflon/composites generally seem to be usable. The fluorocarbons may be marginal at -40°F (-40°C), and, as mentioned above, an increase of compression might be beneficial.

The softer compounds seem to seal slightly better at low temperature, but they are more easily damaged, a factor which affects their usable life.

The Teflon and certain Teflon/composites did well in the low-temperature tests, but higher compression set affects reusability. However, they might be considered for single-use purposes. These seals also failed at 0°F (-18°C) when retested in fixtures with widened grooves in preparation for the high-temperature tests, possibly from lack of lubrication.

All compounds tested at high temperature performed well at their estimated 10-hour life temperature. Once the designer has calculated a reasonable estimate of normal operating temperature, a compound can be selected on the basis of its long-term use temperature, as well as other necessary factors, such as chemical and radiation resistance. The peak high temperature and duration for the hypothetical fire event can then be estimated. If the peak is less than the material's 10-hr rating and the duration is less than 2 hr, the compound should be satisfactory. If not, it will probably be necessary to change compounds to obtain a higher temperature rating.

5.3 Design Test

To support the documentation used in a licensing application, it will probably be necessary for the design agency to conduct performance tests to verify that the particular combination of compound, groove design, thermal, chemical, and radiation environments, and potential physical deformation during the hypothetical accident conditions meets the regulatory requirements.

6.0 SUMMARY AND CONCLUSIONS

6.1 Low-Temperature Seal Performance Testing

The low-temperature test series showed the need for careful material selection to meet RAM packaging criteria. Manufacturer ratings typically relate to criteria significantly different from that required of RAM packaging and therefore should not be relied upon without careful examination. An added concern for low-temperature seal design that was not evaluated here is relative flange movement due to vibration or shock. A very hard or brittle seal would be unlikely to maintain leaktightness if interface surfaces moved.

The majority of materials tested gave the indication of meeting the basic -40°F (-40°C) criterion for RAM packaging. The exceptions were fluorocarbon materials, which failed sufficient tests to be of major concern.

6.2 High-Temperature Seal Performance Testing

Manufacturers' nominal high-temperature ratings apply to an estimated seal life at 1000 hrs. If a package were routinely subjected to a high temperature, as in the case of a high internal heat load, the manufacturer's 1000-hr rating might be a reasonable value for design. However, a hypothetical accident fire is most certainly a singular event of limited exposure time. These tests showed that seal life at elevated temperature estimates can be used to increase the upper limit of elastomeric materials.

While reasonableness must be maintained when applying this philosophy to cask design, a 50 to 90°F (28 to 50°C) increase in usable temperature can greatly aid a designer. It also provides the designer additional options for seal material selection.

As previously noted, seals were stored for a 3- to 6-yr period. High-temperature test results show no apparent degradation of performance due to aging.

No attempt was made to determine if batch-to-batch variations or fixture-induced effects were present in the data. There were too few failures to do these analyses.

6.3 Seal Groove Design

Seals performed well in the groove designs used in this test series. Specific controlled tests were not performed to compare performance in standard groove width fixtures at fixed temperatures to observe whether performance degraded. Groove designs from several seal manufacturers define wider nominal groove widths, presumably with higher temperatures in mind.

Thermal expansion of the seal material at high temperature is an important consideration in groove design. To assure satisfactory performance, the respective volumes of the groove and seal must be compared to verify that the groove does not become overfilled at peak temperature.

7.0 REFERENCES

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Parker Seal Group (O-Ring Division), 1991. "Parker O-Ring Handbook, ORD 570," Lexington, KY 40512.

Warrant, M., and C. Ottinger, 1989. "Compilation of Current Literature on Seals, Closures, and Leakage for Radioactive Material Packagings," SAND88-1015, Albuquerque, NM.

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APPENDIX A

TEST FIXTURE FABRICATION DRAWINGS

These drawings show machining dimensions for the fixtures used in the high-temperature tests (after groove widening). All low-temperature tests were conducted in unmodified fixtures (dash number -000).

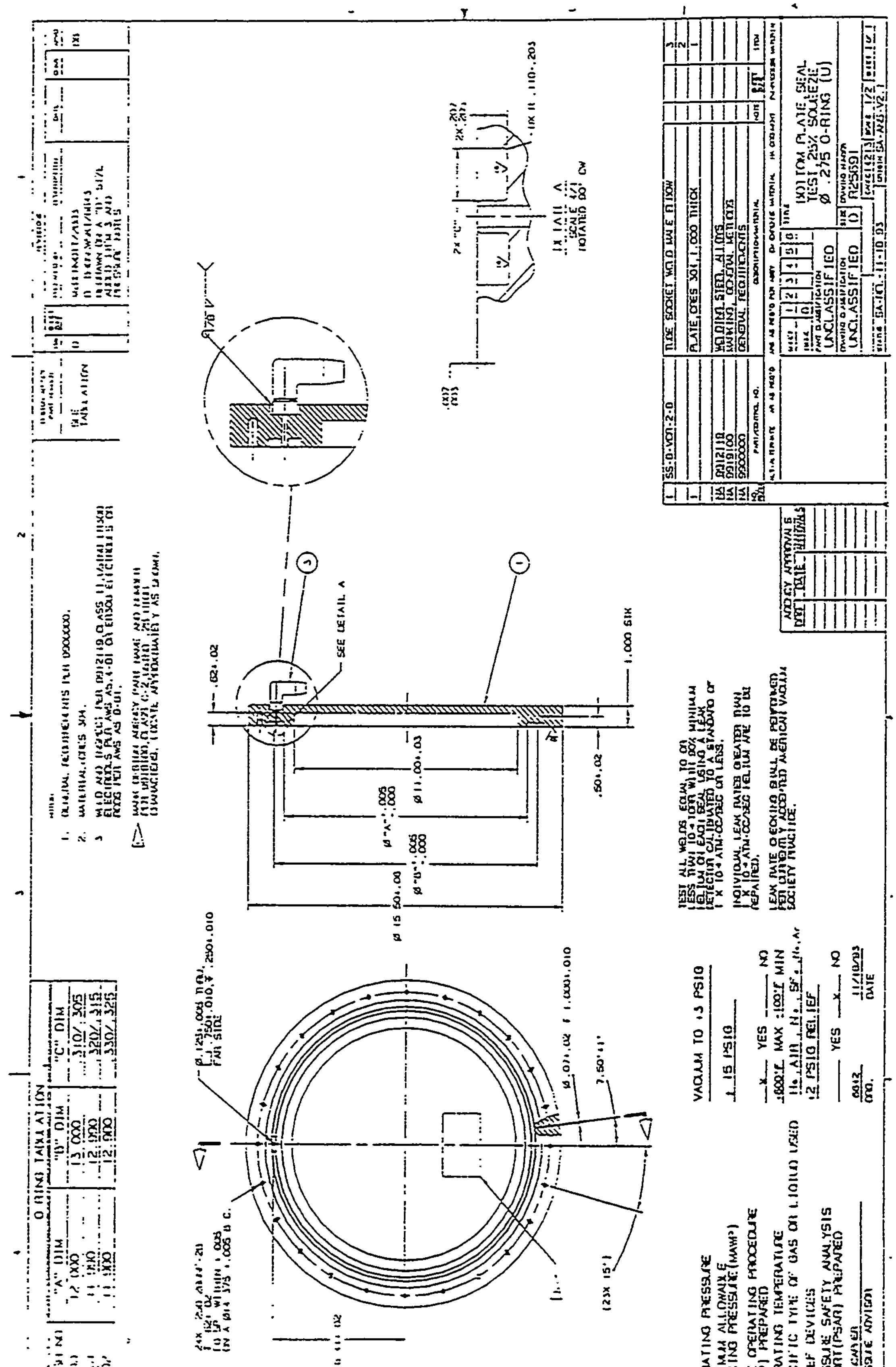


Figure A-1. Seal Performance Test Fixture, Bottom Plate

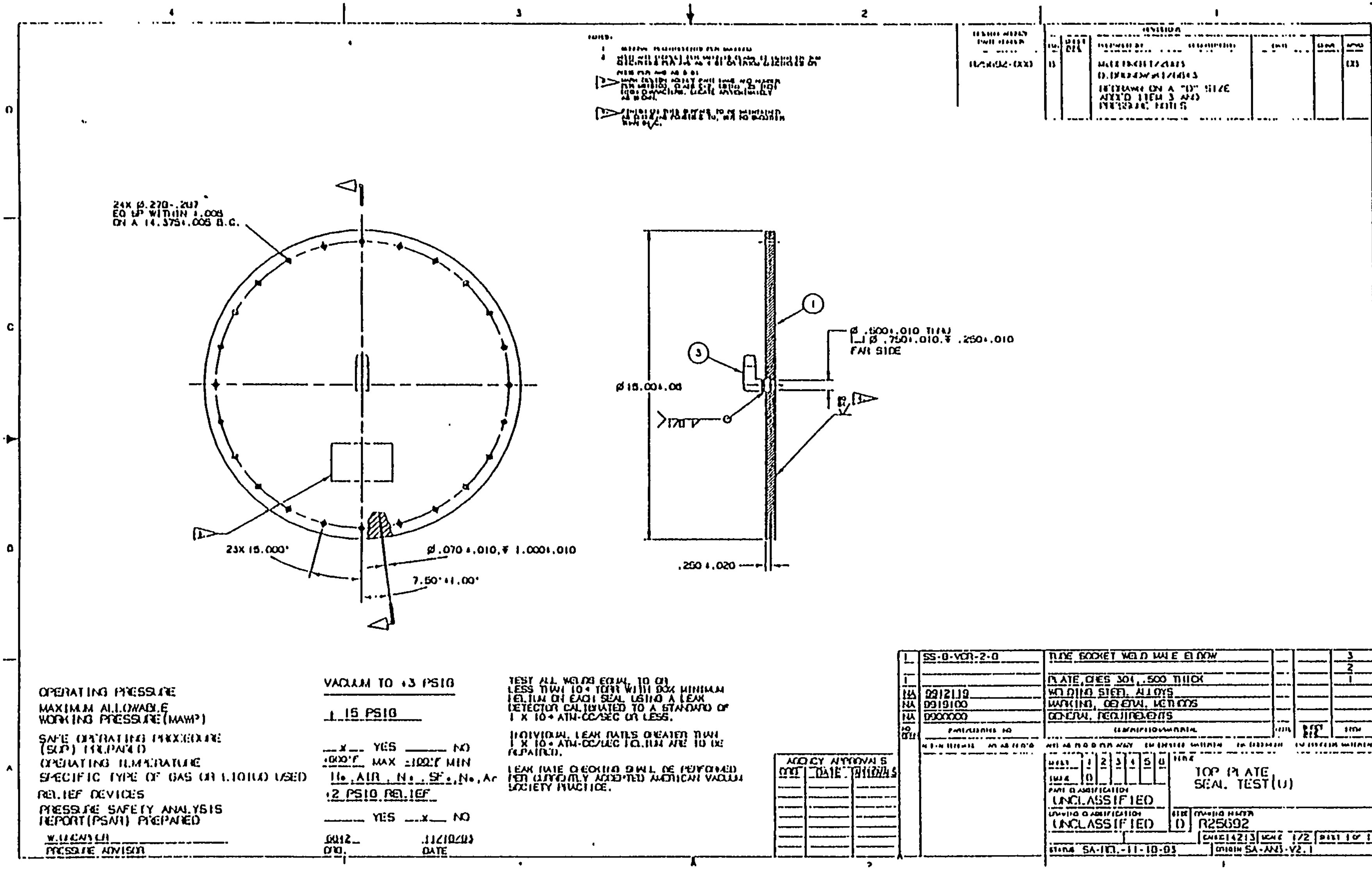


Figure A-2. Seal Performance Test Fixture, Top Plate

APPENDIX B

CIRCUMFERENCE-MEASURING DEVICE DRAWINGS

This appendix contains the drawings showing machining and assembly required for the O-ring circumference-measuring device.

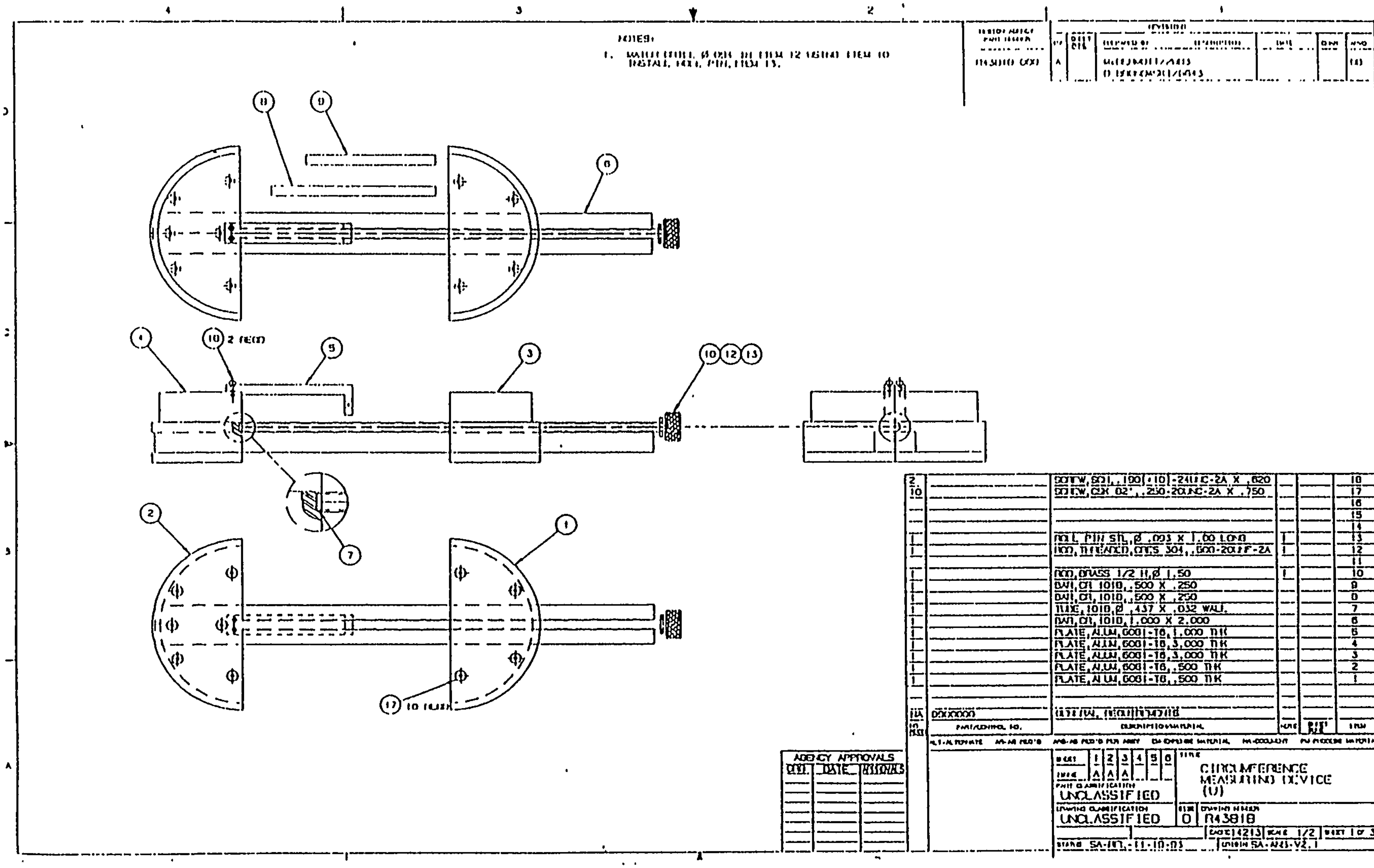


Figure B-1. . Circumference-Measuring Device

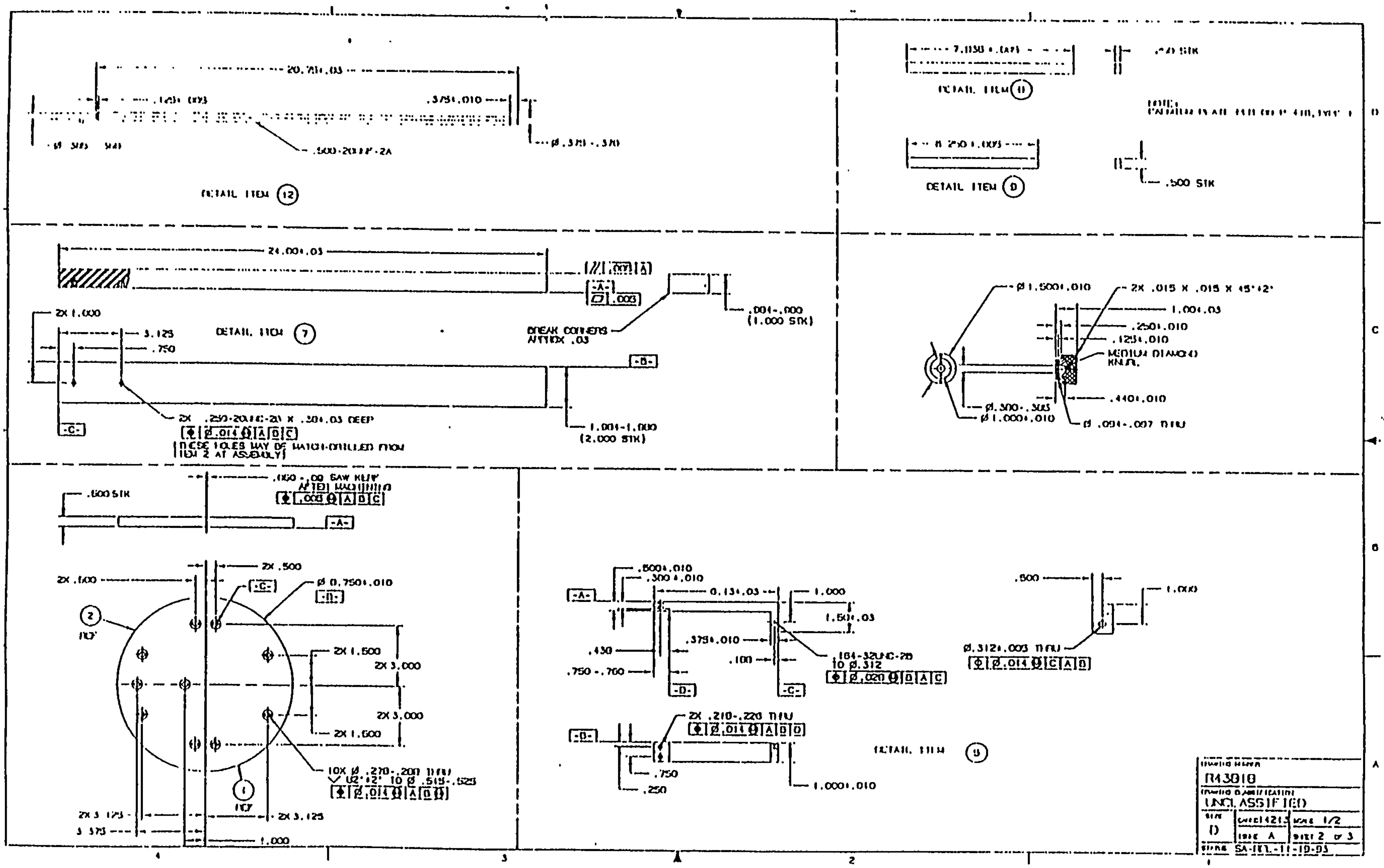


Figure B-2. Circumference-Measuring Device: Machining

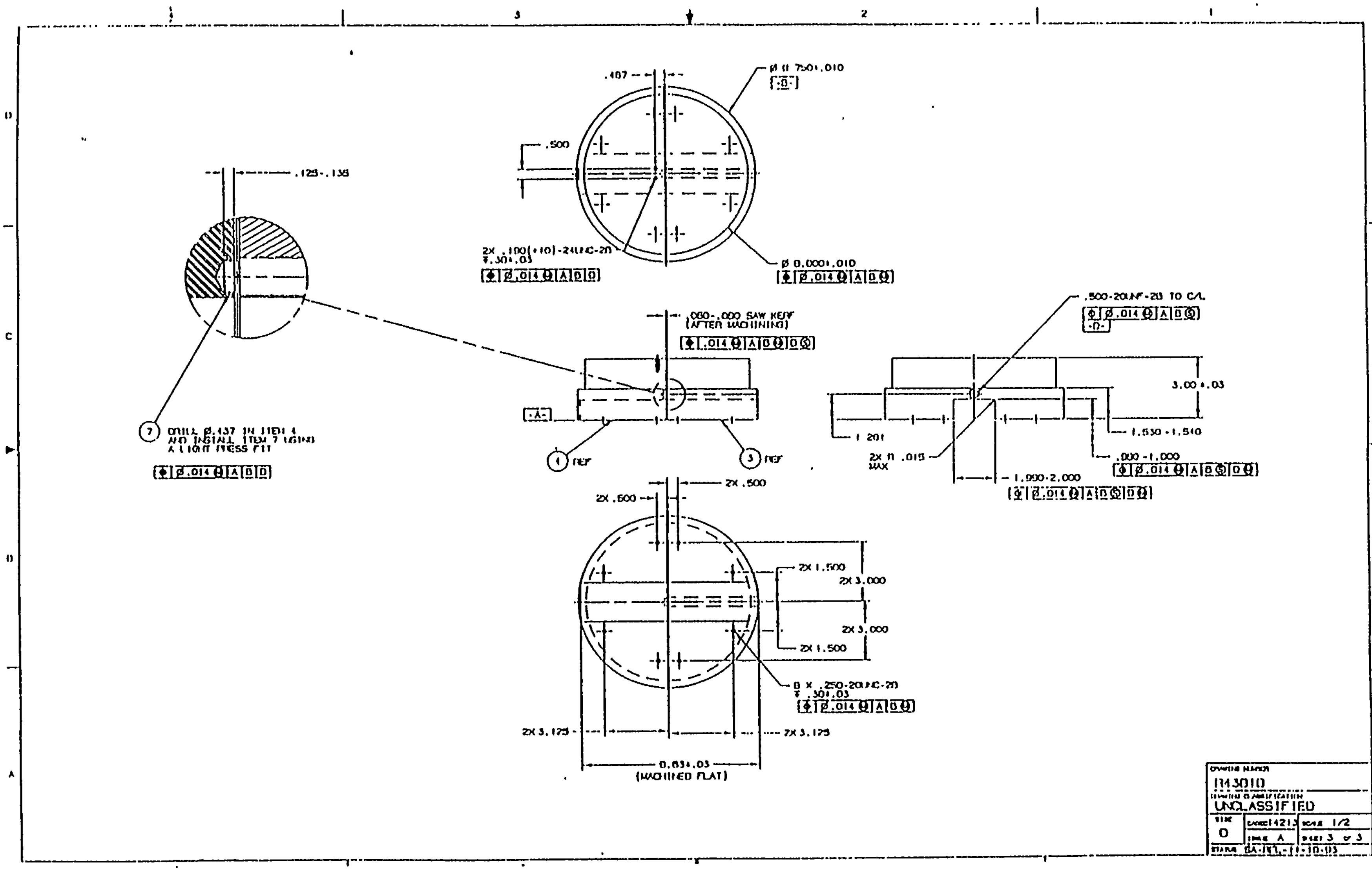


Figure B-3. Circumference-Measuring Device: Assembly

APPENDIX C

LOW-TEMPERATURE SEAL PERFORMANCE TEST PROCEDURE

This appendix contains the reviewed and approved formal procedure for conducting the low-temperature seal performance tests.

April 30, 1990

STP-CTTP-1
Page 1
Rev. A

SANDIA NATIONAL LABORATORIES

TRANSPORTATION SYSTEM DEVELOPMENT DEPARTMENT

SEAL TECHNOLOGY PROGRAM

AUTOMATED CONTROLLED TEMPERATURE SEAL TEST PROCEDURE

Page	1	2	3	4	5	6	7	8	9	10	11	12	13
Revision	A	A	A	A	A	A	A	A	A	A	A	A	A

Prepared by: D. L. Humphreys, Lead Test Coordinator — 6-7-90 Date

K. R. Edwards, Test Coordinator — 6-7-90 Date

Approved by: G. F. Hohnstreiter, Division Supervisor, 6322 — 6/18/90 Date

T. L. Sanders, Division Supervisor, 6323 — 6/13/90 Date

M. M. Madsen, Task Leader — 6/13/90 Date

R. M. Baehr, 6320 QA Coordinator — 6/12/90 Date

Reviewed by: W. L. Uncapher, 6320 Pressure Safety Advisor — 6/12/90 Date

SANDIA NATIONAL LABORATORIES

TRANSPORTATION SYSTEM DEVELOPMENT DEPARTMENT

SEAL TECHNOLOGY PROGRAM

AUTOMATED CONTROLLED TEMPERATURE SEAL TEST PROCEDURE

Issue Summary and Distribution

Issue	Date	Prepared By	Purpose of Issue
A	04/90	D. L. Humphreys	Original Test Procedures

Copy	Revision	Name	Org.	Purpose/Project
1	A	D. L. Humphreys	6322	Seal Test
2	A	G. F. Hohnstreiter	6322	Supervisor
3	A	K. R. Edwards	6323	Seal Test
4	A	M. M. Madsen	6323	Seal Test
5	A	T. L. Sanders	6323	Supervisor
6	A	R. M. Baehr	6320	Quality Assurance Coordinator

SANDIA NATIONAL LABORATORIES

TRANSPORTATION SYSTEM DEVELOPMENT DEPARTMENT

SEAL TECHNOLOGY PROGRAM

AUTOMATED CONTROLLED TEMPERATURE SEAL TEST PROCEDURE

1.0 PURPOSE

This document defines the procedure to be used to measure, assemble, and conduct controlled temperature tests on the Seal Test fixtures.

2.0 SCOPE

This procedure applies to the measuring, assembly, and controlled temperature testing of the Seal Test fixtures by Sandia National Laboratories, Department 6320 personnel.

3.0 RESPONSIBILITIES

Task Leader	M. M. Madsen________________	Org.	6323
Lead Test Coordinator	D. L. Humphreys____	Org.	6322
Test Coordinator	K. R. Edwards__________	Org.	6323
QA Coordinator	R. M. Baehr_____________	Org.	6320QA

Responsibilities of each of the parties listed above are detailed in the Project Quality Assurance Program Plan (PQAPP).

4.0 MEASURMENT OF O-RINGS - PRE AND POST TEST

4.1 Reference Documents
Testing Program Document - Seal Technology Testing

4.2 Equipment Required
Circumference Measuring Device (CMD)
Micrometer
Vernier Caliper
Calculator
Plastic Bags
Tags

4.3 O-ring Measurement Data Records
O-ring measurement data (pre and post test) will be recorded on "O-RING DIMENSIONAL CHECK LIST" data sheets, see page 11.

4.4 Pre/Post Test O-ring Measurements Procedure
Attach a copy of the calibration certificate for the dial caliper and vernier caliper to the "DATA & CHANGE REPORT ATTACHMENT INDEX", see page 12.

1. The new O-rings are tagged with an assigned number, cure date, and batch number written on each tag. This tag will identify, and stay with, the particular O-ring throughout the testing series.

2. The ambient temperature is logged on the "O-RING DIMENSIONAL CHECK LIST" data sheets.

3. The O-ring cross-section is measured at four (4) places approximately 90 degrees apart around the circumference. A 0-1" micrometer is used to measure the cross-section and the results are then logged on the "O-RING DIMENSIONAL CHECK LIST" data sheets.

4. The O-ring is then placed on the Circumference Measuring Device (CMD). The gap of the CMD is then adjusted so that there is no sag in the O-ring. The micrometer is used to measure the cross-section at the gap to insure the O-ring is not being stretched. A 0.001 to 0.003 inch difference is allowed in this measurement. The O-ring is rotated around the CMD and the cross-section is measured again. (See drawing of the CMD fixture on page 6.)

5. The gap distance is measured with a dial caliper and this number is logged on the "O-RING DIMENSIONAL CHECK LIST" data sheets. This number is used in the

formula (shown below) to determine the inside diameter of the O -ring.

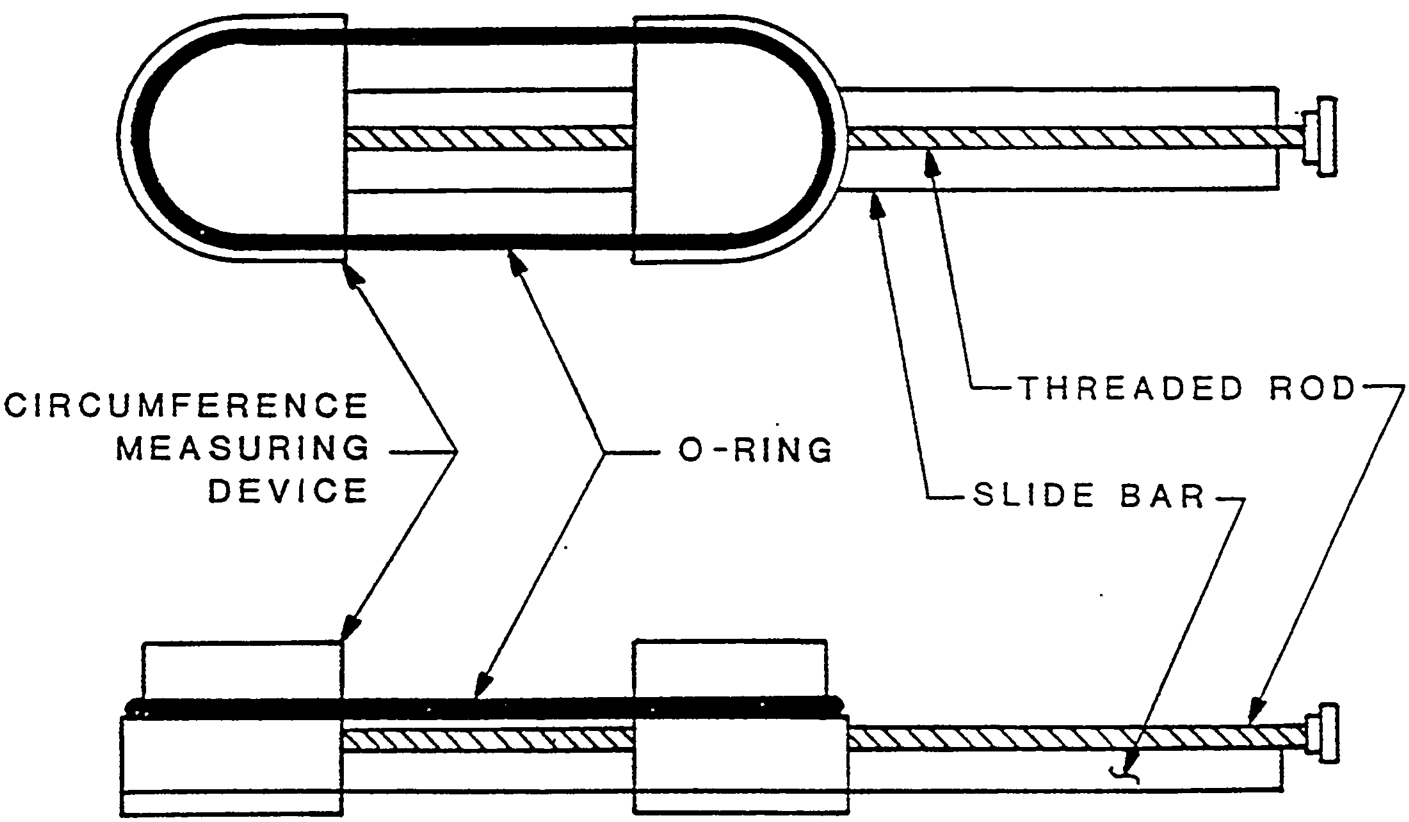
$$\pi D + 2L = C \text{ (Circumference)}$$

$$\pi D = 24.97 \text{ (The fixed diameter of the CMD)}$$

$$C/\pi = \text{Diameter of the O-ring.}$$

6. Repeat steps 1 thru 5 to perform the Post Test measurements.

4.5 Circumference Measuring Device (CMD)



5.0 ASSEMBLY - SEAL TEST FIXTURE

5.1 Reference Documents

Testing Program Document - Seal Technology Testing

5.2 Equipment Required

Seal Test Fixture
Torque Wrench
Assembly hardware (Screws, and washers)
O-ring
Denatured alcohol
Vacuum grease - Dow Corning

5.3 Seal Test Data Records

Test parameters that include test identifiers, compression, and surface finish will be recorded on computer printouts. Additional test data included on the computer printout are fixture top and bottom plate temperatures, chamber temperature, and leak rate as a function of time. Following completion of the permeation tests, leak rate will be plotted vs. time. Temperature vs. leak rate will be plotted for temperature variation tests. The computer data will be stored on a floppy disk at the conclusion of each test and placed in the quality assurance files at the conclusion of this activity.

5.4 Assembly Procedure

Attach a copy of the calibration certificate for the torque wrench to the "DATA & CHANGE REPORT ATTACHMENT INDEX", see page 12.

1. Clean the Top Fixture Plate to be tested thoroughly with alcohol and dry.

2. Clean the Bottom Fixture Plate to be tested thoroughly with alcohol and dry.

3. Clean the inner and outer O-rings to be tested thoroughly with alcohol and dry.

4. Examine all mating surfaces of the top and bottom plates and the O-rings for damage and contamination.

5. Grease the O-rings with a small amount of vacuum grease and insert into the O-ring grooves of the fixture plate.

6. Assemble the Top and Bottom Fixture Plates using the 1/4-28 x 1.00" long screws and washers if required.

7. Torque the 1/4-28 x 1.00" long screws uniformly to 100 in. lb.

6.0 CONTROLLED TEMPERATURE TEST PROCEDURE - SEAL TEST FIXTURE

6.1 Reference Documents
Testing Program Document - Seal Technology Testing

6.2 Equipment Required
Mass Spectrometer Leak Detector - Alcatel (Model ASM-51)
Environmental Chamber - Tenny Six or Blue M
Calibrated Leak - Varian (Model 0981-F8473-301)
Thermocouples - Type K
Helium bottle and regulator
Absolute Pressure Gage - Wallace & Tiernan (Model 61B-1A-0300)
Barometric Pressure Gage - Baratron MKS (Model PDR-D-1)
Vacuum Pump - Welch

6.3 Temperature Test Procedure
Attach a copy of the calibration certificates for the Leak standard, thermocouples, Data Control Unit, and absolute pressure gage to the "DATA & CHANGE REPORT ATTACHMENT INDEX", see page 12.

1. Place the Seal Test Fixture on the fixture rack in the temperature chamber, and attach the vacuum hose connections.

2. Insert the thermocouples into the top and bottom plates of the Seal Test Fixture.

3. Set target temperature on the temperature chamber per the manufacturer's instructions.

4. Turn on the Alcatel Helium Leak Detector and, after LN is added, allow to warm up for 30 minutes.

5. Turn on Hewlett Packard computer. Login and type in the password. Follow the prompts for "START A TEST", where the O-ring material type and size is entered in the file.

6. Use softkey "STD LEAK ON" to open valving needed to calibrate the Alcatel Leak Detector, then proceed with calibration per manufacturer's instructions.

7. Turn on the vacuum pump.

8. Turn on the temperature chamber.

9. Open the valve on the helium bottle.

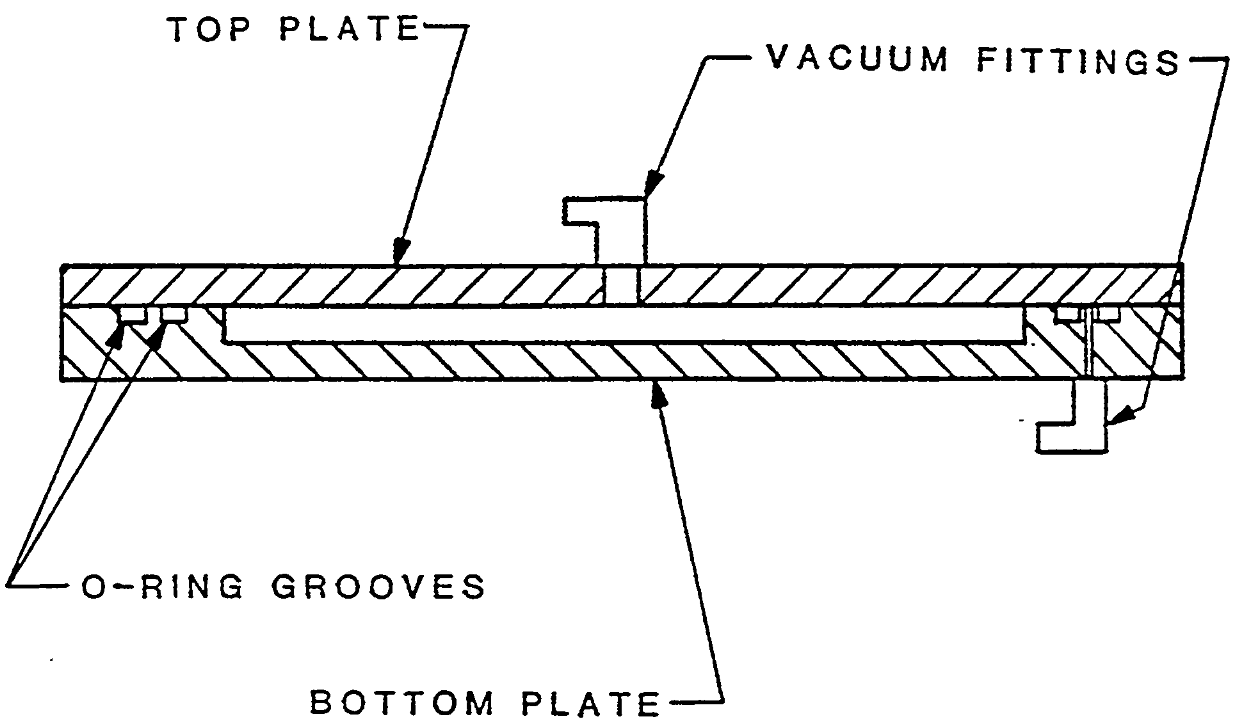
10. When the target temperature is reached, select "TESTING" mode and open helium valve for five (5) seconds. (This is five times longer than it takes the Alcatel to detect helium, but not long enough to allow for permeation.)

11. Having survived the target temperature, the temperature is changed, as required, until a criterion, such as loss of high vacuum at 1.0E-04 atm cc/sec, is met.

12. Upon ending all tests, plot the data, and printout a copy of the Leak Test data which is to be put into the Data Record book. In addition, write a brief synopsis of the test results in the Seal Test Log book.

13. After allowing the fixtures to return to ambient temperature, remove the fixtures from the temperature chamber, and disassemble, carefully noting any observations on the computer printout. Photograph any unusual observations.

7.0 SEAL TEST FIXTURE CONFIGURATION



8.0 O-RING DIMENSIONAL CHECK LIST DATA SHEET

O-RING DIMENSIONAL CHECK LIST

Material:______________________________ Date:__________

Manufacturer: Parker Seal Company

Batch Number:______________________________ Cure Date:__________

Test Conducted By:______________________________

PRE TEST:

Gap Length (L):______________________________

Inner Circumference:______________________________

Inside Diameter:______________________________

Cross Sectional Diameter at Position 1:______________

at Position 2:______________

at Position 3:______________

at Position 4:______________

Temperature:______________ Degrees F.

POST TEST: Date:__________

Gap Length (L):______________________________

Inner Circumference:______________________________

Inside Diameter:______________________________

Cross Sectional Diameter at Position 1:______________

at Position 2:______________

at Position 3:______________

at Position 4:______________

Temperature:______________ Degrees F.

9.0 DATA & CHANGE REPORT ATTACHMENT INDEX

SEAL TECHNOLOGY PROGRAM
PROJECT QUALITY ASSURANCE PROGRAM PLAN

TABLE 1

Attachment No.	Document Type	Designation (if applicable)	No. of Pages

10.0 STP PROCEDURE CHANGE REPORT

Change No._________ Page _____ of _____

STP PROCEDURE CHANGE REPORT

Procedure Title____

Procedure No.______________ Applicable Test Number(s)___________

Procedure Page_____________ Applicable Unit Number(s)___________

Procedure Rev. No._________ Date___________

Change ____

Reason for Change ___________________________________

Comments ____

Approved By:

Task Leader	Date	Q. A. Coordinator	Date

Other Approvals

Title	Date	Title	Date

10.0 STP PROCEDURE CHANGE REPORT

Change No. -101 Page 1 of 4

STP PROCEDURE CHANGE REPORT

Procedure Title CONTROLLED TEMP TEST PROCEDURE
Procedure No. STP-CTTP-1 Applicable Test Number(s) 0046 THRU
Procedure Page ______ Applicable Unit Number(s) NA
Procedure Rev. No. A Date 9-7-93

Change SEE ATTACHED SHEETS

Reason for Change

Comments

Approved By:

[signature] 9/9/93
Task Leader Date Q. A. Coordinator Date

Other Approvals

W.B. [signature] 9/8/93
Title Date Title Date
TECH. DIRECTOR

1.0 Introduction

This change report is to revise several parts of Controlled Temperature Test Procedure STP-CTTP-1, to allow the procedure to be used for an additional/separate series of low temperature leak tests. Specific changes, additions, and deletions are listed in Section 6 below.

The Quality Assurance level for the test series is designated as QL-3, Minor.

2.0 Purpose/General Information

The purpose of this test series is to evaluate the effects of widened O-ring grooves on seal performance at reduced temperatures. It has been determined through proof-of-principal/scoping tests and through literature searches that widened grooves are necessary to obtain satisfactory seal performance at significantly elevated temperatures. The widened grooves allow room for the seal material to expand when heated to high temperatures, i.e., above 300°F. Since a package/cask can have only a single groove design, evaluation is required to determine if the widened grooves adversely affect seal performance at reduced temperatures where the wide groove is neither necessary nor desired.

3.0 New Test Series Description

The test series will be an abbreviated series consisting of reduced temperature tests on selected materials. Materials have been selected based on their performance in the previously performed tests; materials which did not maintain a seal at -40°F were not considered for this series. One test will be performed on each material/batch number as listed below. The results of these tests will be compared against prior test data. Multiple tests of specific materials and/or batch numbers will not be performed unless data is inconclusive and requires further evaluation. Materials which perform satisfactorily during this series will then be evaluated in future high temperature tests.

3.1 Materials

Seal Compound	Batch Numbers			Fixture Type
C0873-70	0800905	0809701	0810949	B
C1124-70	0809701	285844		B
E0540-80	801255	0810976		B
E0740-75	0791171	0810101	0810976	B

3.1 Materials - continued

Seal Compound	Batch Numbers			Fixture Type
F0953-70	289590	292539	294683	A
L0677-70	69700	70719	70762	A
R1801-70	17132	17138		A
S0383-70	69633	70423		A
S0604-70	69382	70141		A
S0613-60	69679	70553		A
S0899-50	69853	70663		A
V0747-75	277185	285290		A
V0835-75	289464	291944		A
W19657-GLT	193			A

4.0 Test Fixtures

Test fixtures have been modified with the widened grooves. One set of three fixtures (designated as 1A, 2A, & 3A) have grooves that are .020 inch wider than standard Parker groove design dimensions. A second set of three fixtures (1B, 2B, & 3B) have grooves that are .010 inch wider than standard design. Fixture utilization is listed above and has been selected according to material type and its expected upper temperature limit.

5.0 Changes to Procedure STP-CTTP-1

Significant changes to the existing test procedure are as follows;

5.1. Procedure Section 3.0. Responsible persons are:
Task Leader - P. McConnel, 6643
Leak Test Coordinator - W. Leisher, 6643
Test Technician - D. Bronowski, 6643
Quality Assurance - L. Martin, 6600

5.2 Procedure Section 4.0. No dimensional pre- or posttest inspections will be performed on the O-rings.

5.3 Procedure Section 5.0. Fixtures will be assembled without the use of vacuum grease. Fixture bolt torque is lowered from 100 in-lb to 75 in-lb. Data records, i.e., seal and fixture identification and related data will be entered directly into the test data base of the Seal Software Program.

5.4 Procedure Section 6.0. The assembled fixtures will be installed in the temperature chamber and their respective plumbing fittings and thermocouples connected and verified. An assembly verification leak test will be performed on each fixture at ambient temperature. After the fixtures as shown to be acceptable, i.e., leakage rates less than 1E-8 cc/s, the chamber will be used to cool the fixtures to 0°F. The fixtures will then be leak tested at this temperature. Fixtures will next be cooled to -10°F, -20°F, -30°F, -40°F, -50°F, and -60°F, with a leak test being performed at each temperature step. Each fixture will continue to be tested as long as the detector can maintain high-vac/test mode or -60°F is reached. Fixtures may be tested simultaneously as long as the sum of the background readings aren't so high that they could obscure an E-7 cc/s leakage rate.

After the final low temperature step, the fixtures will be allowed to warm to ambient temperature over approximately 2 to 3 hours. A final, post test leak test will be performed at ambient temperature.

All references to data acquisition commands and keystrokes are deleted as a new/different data acquisition system and control software will be utilized.

APPENDIX D

STATISTICAL ANALYSIS OF LOW-TEMPERATURE LEAK TEST DATA

This work was performed by the SNL Human Factors and Statistics Department to determine the probability of satisfactory sealing function at -40°F (-40°C) for specific compounds.

Sandia National Laboratories

Albuquerque, New Mexico 87185

date: October 30, 1992

to: W. B. Leisher, 6643

from: Brian Rutherford, 323

subject: Statistical Analysis of O-Ring Test Data

I. Introduction and Summary

The Department of Energy has requested Sandia National Laboratories Transportation Development Department, 6643, to evaluate o-ring compounds in order to determine which ones are best suited for application in nuclear waste shipment. In order to make this evaluation, a series of tests were planned on small o-rings to identify those compounds that could be considered reasonable candidates for cask applications. To date, these tests have focused on o-ring performance at low temperatures. In these tests, the temperature is lowered for specimen o-rings of different compounds until failure (excess leakage) occurs.

The low temperature failure data from these tests indicate that elements of the testing equipment are affecting the results. Specifically, the results indicate that at least one of the flanges used to secure the o-ring during testing is leading to appreciably higher failure temperatures than are the other flanges. There are also substantial differences among o-ring compounds in low temperature performance with estimated average failure temperatures ranging from -83°F to 11°F. Details pertaining to these results are given in the following sections.

The purposes of this memorandum are to report the results of my statistical analysis investigating the effect of testing equipment and to give a preliminary report of available test results for 27 o-ring compounds. The memorandum also provides limited guidance toward future testing plans. The next section gives a brief description of the data and of the experimental equipment. The third section describes the statistical analyses and results.

II. Data and Testing Equipment

Several candidate o-ring compounds were selected based on their use in similar applications or at the recommendation of seal manufacturers. Table 1 lists the o-ring compounds included in the initial tests.

Each test involves compressing the o-ring between a pair of plates, or flanges, and filling the inner volume with helium. The amount of helium to escape to the outside of the o-ring is measured by a calibrated mass spectrometer. Each o-ring is monitored continuously at temperatures starting at 20°F and reduced until a failure of the o-ring occurs. A failure is considered to occur when the leak rate is measured to exceed 10^{-5} cc/sec. Leak rates are recorded at 10°F increments and the failure temperature is recorded. Only the failure temperatures were used in this statistical analysis.

Several samples of each o-ring were tested with different flange pairs, with some pairs used more than once. Flanges included for the present tests were 1A, 1B, 2A, 2B, 3A, 3B, K1, K2, K3, and T6. In general, the flanges were used in pairs and the bottom flange had the same identification as the top flange. Exceptions were: The bottom flange associated with T6 was labeled B1; and the 6 tests with top plates K1, K2, or K3 were matched with bottom plates 1B, 2B, and 3B respectively. This procedure introduced a small amount of correlation into the results for the associated flange pairs; however only top flange identification was considered in the statistical analysis described in Section III because so few of the tests were affected.

A total of 237 tests were reported; of these, 4 tests yielded censored results (the tests were stopped prior to failure for reasons not related to the variables considered in the analysis). A complete listing of the data is given as an appendix. The four censored results are circled.

III. Statistical Analysis and Results

The statistical analysis of the o-ring data focused on only a few of the possible variables that might influence the failure temperature. The variables considered in the analysis include o-ring compound, flange, and possible interaction between the o-rings and the flanges. A significant interaction might indicate that some o-rings are better at adapting to rough, smooth, or scratched surfaces than others.

At least one potentially important variable, o-ring batch, was not part of the test and thus has not been included in the analysis because most o-rings were obtained from the same batch for each supplier. Future samples should include o-rings from different batches to evaluate batch-to-batch variability of failure temperatures and to assure that this (possibly influential) variable is accounted for when evaluating the expected failure temperatures.

To compare o-ring compounds and the flange pairs, a statistical analysis was performed using SAS (1985) Procedures GLM. The standard ANOVA was performed to check for o-ring or flange pair affects and to investigate the possibility of an o-ring-flange interaction. For this portion of the analysis, the 4 censored values were replaced by their conditional expected value (determined iteratively). The assumptions required for this type of analysis to be appropriate and techniques that help determine whether or not they are appropriate are discussed in most introductory texts on analysis of variance or experimental design and briefly in SAS (1985). From an investigation of the failure temperature data, it appeared that these assumptions were not violated. One assumption, that of equal variances within each o-ring-flange pair combination, could not be tested because of the limited number of tests in most of these combinations.

Table 2 shows the results of this portion of the analyses. The significants of any factor or interaction is indicated by the p-value which specifies the probability of observing a value of the test statistic (specified by the F value) as high or higher than that observed by chance. The value .45 corresponding to the interaction indicates that the interaction term is not affecting failure temperatures. This means that differences between flanges are consistent across the o-ring compounds. Table 2 also indicates that differences among o-rings and flanges are both quite significant. A second analysis via SAS procedure LIFEREG, which can accommodate censored data (but not a model that includes interactions) supports this conclusion.

Different o-ring compounds were tested with (different sets of) flange pairs. Thus, any direct comparison of the data from different o-ring compounds is influenced by (or biased by) differences among flanges. The data permit, however, the separation of these influences through the calculation of "least squares means" which estimate the effect of flange pairs and adjust the o-ring mean estimate to accommodate these differences. Searle, et al, (1980) or SAS (1985) give details of how these adjustments are made. Least squares means can also be calculated for flange pairs. Figure 1 gives these means and shows the magnitude of the differences between flanges; there is a range of some 70°F across the 10 sets of flanges used in these tests. This means that failure temperatures obtained from one set of flanges may not be at all representative of an o-ring compound's capability.

Any flange contributing to relatively high (warm) failure temperatures should be reworked or excluded from further testing because its inclusion in the experiment will only detract from the ability to discriminate between o-ring compounds. To facilitate the preliminary comparison of o-ring compounds, I dropped several flange pairs from the analysis. Table 3 provides a list of the number of samples (n) tested by each flange and their least squares means. The four flanges that saw very limited use (a total of 8 tests for K1, K2, K3 and T6) also provided the extreme least squares means. Since these were used in so few tests, their results were not included for the o-ring comparisons to prevent the possibility of introducing a bias. In addition, flange 1B appears to perform significantly worse than the remaining flanges. Results using this flange, were also eliminated from data set for the o-ring comparison. Note that the least squares means for the remaining flanges have a range of only 12°F.

To compare o-ring compounds, several statistics were computed using the data set with the omissions described above. Table 4 gives a listing of the compounds and several of these statistics. The first column gives the number of samples (n) tested of each compound. The second and third columns of Table 4 give the mean and standard deviation of the recorded failure temperatures (across n samples). The standard deviation reflects the variance among the reduced set of flanges as well as o-ring compound variability. The fourth column gives the least squares means. Figure 2 provides a plot of these means. Again, a wide range of estimates are observed from -70°F to 15°F. The values in parenthesis in Column 4 show the least squares means computed using the entire data set. Note the differences in estimates are substantial when the flanges that appeared to yield higher (warmer) failure temperatures are included. To get a realistic estimate of the failure temperatures to expect based on the actual use surface conditions, one must compare the anticipated use surfaces to surfaces of the flange pairs used in this set of experiments. All flange surfaces in the reduced set were milled to 64 microinches RMS.

The fifth column of Table 4 provides an estimate of the probability of not failing at -40°F for each compound. Survival at -40°F is part of a Nuclear Regulatory Commission requirement on o-ring performance. These survival probability estimates and the confidence bounds given in the final two columns of Table 4 are based on the assumption of normally distributed o-ring failures for o-rings tested using any specific flange and the assumptions of normally distributed flange effects and a random selection of flanges is required for these probabilities and confidence bounds. Note that there is a spread in the estimated survival probabilities from 0 to 100 percent across the different compounds.

The final two columns of Table 4 provide an indication of the uncertainty associated with the estimates in Column 5. Column 6 gives a lower 95 percent bound and Column 7 gives an upper 95 percent bound on the survival probabilities for each o-ring compound at -40°F. The widths of the confidence intervals demonstrate the fairly high level of uncertainty associated with the estimates in Column 5 primarily because of the small sample sizes. The 90 percent bounds are exact for o-ring compounds without censored observations. The technique for their computation is based on the noncentral t distribution. The details and required tables are given in Owen (1968). Creavey-Teflon, NPTFE, and S0383-70 contained censored observations. The confidence bounds for these compounds are approximate as they are based on asymptotic properties of maximum likelihood estimates for percentiles of the failure distribution. A brief description can be found in the SAS (1985) description of procedure LIFEREG.

Should you have questions on any portion of this analysis, or if I can help in planning or analyzing further phases of this evaluation, please call me at 4-3120.

References:

D. B. Owen (1968), "A Survey of Properties and Applications of the Noncentral t-Distribution", Technometrics, Vol. 10, No. 3.

SAS (1985), "SAS User's Guide: Statistics, Version 5 Edition", SAS Institute Inc., Cary, North Carolina.

S. S. Searle, F. M. Speed, and G. A. Milliken (1980), Population Marginal Means in the Liner Model: An Alternative to Least Squares Means", The American Statistician, Vol. 34, No. 4.

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Table 1. O-ring Compounds Included in the Initial Phase of Testing.

19657-GLT
B612-70
C/G-TEFLON-SILICONE
C/G-TEFLON-VITON
C1124-70
C873-70
CREAVEY-ASTRO-SS
CREAVEY-TEFLON
E540-80
E740-75
EYPEL-1801-70
F0953-70
KALREZ 2
L0677-70
L0677-75
NPTFE
R-0404-50
R-0404-70
R1429-70
ROW-TEFLON-SILICONE
ROW-TEFLON-VITON
S0383-70
S604-70
S613-60
S899-50
V747-75
V835-75

Table 2. Analysis of Variance Table for Failure Temperatures. Censored results were replaced by their conditional expected value.

Source	df	Sum of Squares	Mean Square	F value	p-value
O-ring	26	87020.	3347.	10.30	0.0001
Flange	9	18365.	2041.	6.33	0.0001
O-ring*Flange	69	22740.	330.	1.02	0.45
Error	.32	42519.	332.		

Table 3. Least Squares Means for Flange Pairs. Censored results have been replaced by their conditional expected value.

Flange	n	Least Squares Mean
T6	2	-64.
2A	37	-56.
3B	39	-49.
2B	38	-49.
3A	36	-46.
1A	61	-44.
K3	2	-29.
1B	18	-12.
K1	2	-7.
K2	2	6.

Table 4. O-ring Compound Summary Statistics. For each o-ring compound, this table provides the number of tests, mean failure temperature, standard deviation of the failure temperature, adjusted mean failure temperature, estimated survival probabilities, and confidence bounds for the survival probabilities at -40°F. The "*" indicates one or two censored observations were reported for that o-ring compound. The first set of adjusted means in parentheses are based on the entire data set. The remaining statistics are based on the reduced data set.

O-ring Compound	n	Sample Mean (°F)	Standard Deviation (°F)	Adjusted Means (°F)		Estimated Survival Probability	90% Confidence Bounds	
S899-50	11	-84	14.7	(-70)	-83	1.00	.96	1.00
EYPEL-1801-70	3	-73	11.5	(-60)	-73	1.00	.66	1.00
F0953-70	12	-70	13.4	(-54)	-70	.99	.90	1.00
S613-60	15	-70	0.5	(-57)	-70	1.00	1.00	1.00
B612-70	12	-68	23.2	(-55)	-69	.88	.71	.96
L0677-75	6	-69	19.1	(-55)	-68	.93	.58	.99
R-0404-50	6	-63	7.9	(-51)	-65	1.00	.89	1.00
*NPTFE	3	-63	36.1	(-58)	-63	.79	.35	1.00
L0677-70	2	-62	13.4	(-43)	-62	.95	.18	1.00
*CREAVEY-TEFLON	16	-61	33.6	(-44)	-61	.73	.58	.88
CREAVEY-ASTRO-SS	5	-60	18.1	(-47)	-60	.86	.52	.98
E740-75	6	-59	12.2	(-45)	-58	.94	.68	1.00
*S0383-70	6	-57	26.4	(-44)	-58	.76	.51	.96
R-0404-70	6	-55	9.9	(-42)	-57	.93	.67	.99
C/G-TEFLON-SILIC	6	-54	6.8	(-42)	-56	.98	.78	1.00
C1124-70	12	-52	10.3	(-38)	-51	.88	.70	.96
ROW-TEFLON-SILIC	6	-49	8.4	(-37)	-51	.86	.57	.97
S604-70	6	-49	21.0	(-33)	-50	.66	.36	.88
E540-80	8	-41	17.0	(-30)	-41	.52	.29	.75
ROW-TEFLON-VITON	6	-39	19.4	(-26)	-40	.48	.24	.73
C873-70	6	-34	4.6	(-20)	-34	.10	.01	.37
19657-GLT	7	-27	5.3	(-14)	-28	.01	0.00	.14
V835-75	21	-25	18.5	(-12)	-25	.19	.11	.36
R1429-70	6	-16	13.6	(-4)	-18	.04	0.00	.27
C/G-TEFLON-VITON	9	-11	16.3	(-1)	-13	.04	0.00	.20
V747-75	6	-2	11.7	(12)	-1	0.00	0.00	.08
KALREZ 2	3	-13	5.8	(19)	11	0.00	0.00	.21

Appendix. Data listing

Figure 1. Least Squares Means Plotted for Flange Pairs. Results obtained using the circled flanges were not included in the comparison of o-ring compounds.

Figure 2. Least Squares Means Plotted for O-ring Compounds. The plotted means are based on the reduced set of flange pairs. The ordering of the plotted points is listed above the plot.

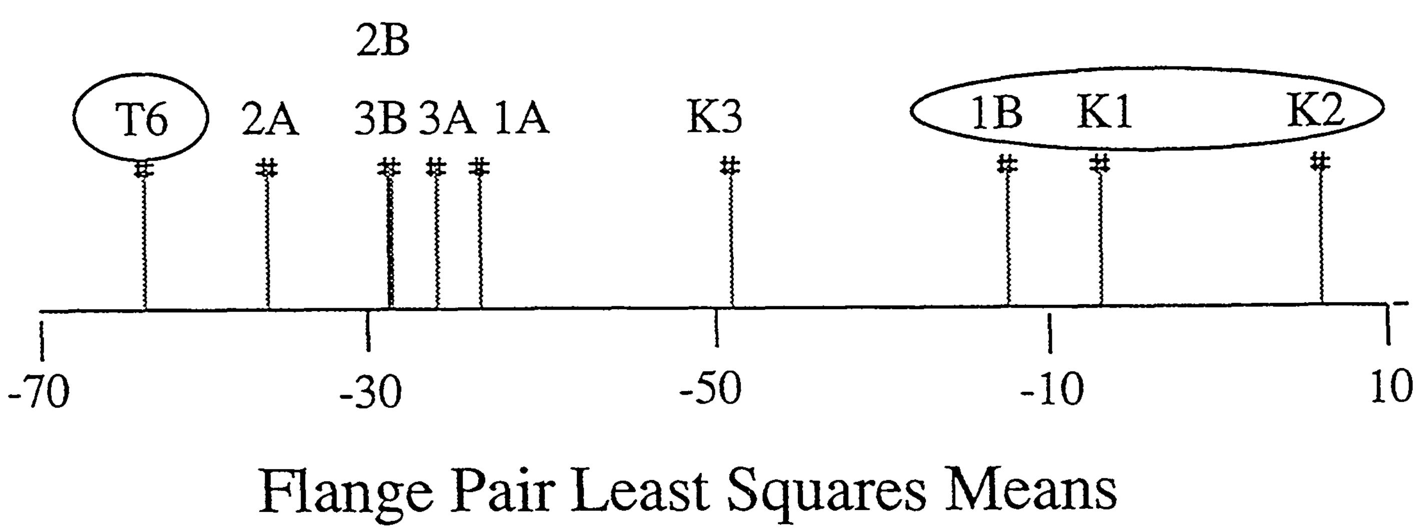

Figure 1. Least Squares Means Plotted for Flange Pairs. Results obtained using the circled flanges were not included in the comparison of o-ring compounds.

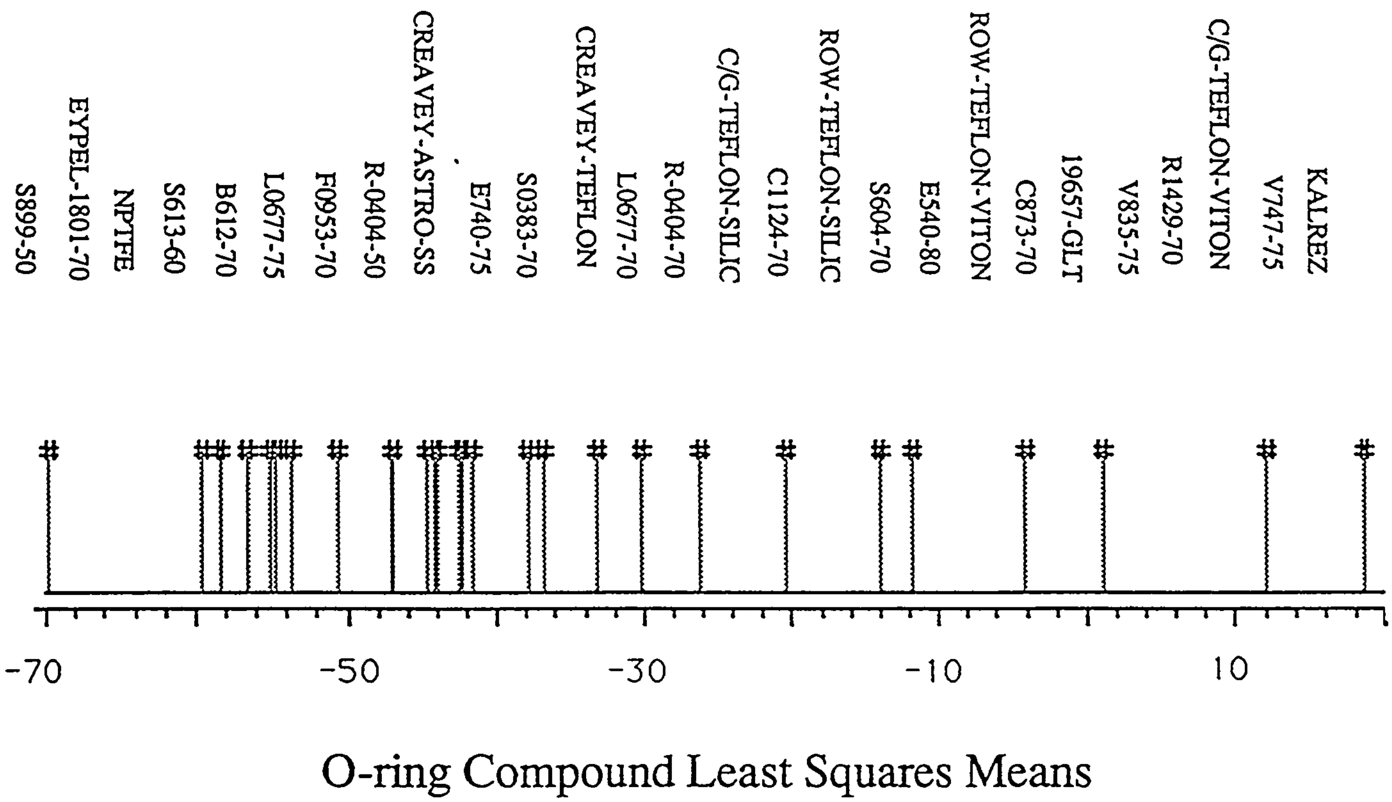

Figure 2. Least Squares Means Plotted for O-ring Compounds. The plotted means are based on the reduced set of flange pairs. The ordering of the plotted points is listed above the plot.

Appendix. Data listing

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t397	19657-GLT	3B	3B	-31
11t398	19657-GLT	1A	1A	-28
11t399	19657-GLT	2B	2B	-29
11t400	19657-GLT	3B	3B	-30
11t401	19657-GLT	1A	1A	-19
11t402	19657-GLT	2B	2B	-19
11t403	19657-GLT	3B	3B	-30
11t169	B612-70	1A	1A	-10
11t170	B612-70	2A	2A	-81
11t171	B612-70	3A	3A	-83
11t178	B612-70	1A	1A	-80
11t179	B612-70	2A	2A	-80
11t180	B612-70	3A	3A	-31
11t347	B612-70	1A	1A	-83
11t348	B612-70	2B	2B	-71
11t349	B612-70	3B	3B	-70
11t350	B612-70	1A	1A	-79
11t351	B612-70	2B	2B	-80
11t352	B612-70	3B	3B	-70
11t386	C/G-TEFLON-SILICONE	1A	1A	-49
11t387	C/G-TEFLON-SILICONE	2B	2B	-60
11t388	C/G-TEFLON-SILICONE	3B	3B	-50
11t407	C/G-TEFLON-SILICONE	1A	1A	-65
11t408	C/G-TEFLON-SILICONE	2B	2B	-50
11t409	C/G-TEFLON-SILICONE	3B	3B	-50
1ap431	C/G-TEFLON-VITON	1A	1A	-30
1ap432	C/G-TEFLON-VITON	2B	2B	-30
1ap433	C/G-TEFLON-VITON	3B	3B	0
11t434	C/G-TEFLON-VITON	1A	1A	-31
11t435	C/G-TEFLON-VITON	2B	2B	10
11t436	C/G-TEFLON-VITON	3B	3B	0
11t437	C/G-TEFLON-VITON	1A	1A	0
11t438	C/G-TEFLON-VITON	2B	2B	0
11t439	C/G-TEFLON-VITON	3B	3B	-20
11t136	C1124-70	1A	1A	-60
11t137	C1124-70	2A	2A	-51
11t138	C1124-70	3A	3A	-40
11t139	C1124-70	1A	1A	-50

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t140	C1124-70	2A	2A	-71
11t141	C1124-70	3A	3A	-50
11t142	C1124-70	1A	1A	-50
11t143	C1124-70	2A	2A	-60
11t144	C1124-70	3A	3A	-50
11t145	C1124-70	1A	1A	-36
11t146	C1124-70	2A	2A	-63
11t147	C1124-70	3A	3A	-40
11t208	C873-70	1A	1A	-30
11t209	C873-70	2A	2A	-39
11t210	C873-70	3A	3A	-41
11t214	C873-70	1A	1A	-32
11t215	C873-70	2A	2A	-32
11t216	C873-70	3A	3A	-31
11t249	CREAVEY-ASTRO-SS	1A	1A	-40
11t250	CREAVEY-ASTRO-SS	2A	2A	-75
11t251	CREAVEY-ASTRO-SS	3A	3A	-60
11t278	CREAVEY-ASTRO-SS	1B	1B	-31
11t279	CREAVEY-ASTRO-SS	2B	2B	-80
11t280	CREAVEY-ASTRO-SS	3B	3B	-43
11t246	CREAVEY-TEFLON	1A	1A	-24
11t247	CREAVEY-TEFLON	2A	2A	-88
11t248	CREAVEY-TEFLON	3A	3A	-10
11t252	CREAVEY-TEFLON	1A	1A	-20
11t253	CREAVEY-TEFLON	2A	2A	-50
11t254	CREAVEY-TEFLON	3A	3A	-86
11t272	CREAVEY-TEFLON	K1	1B	-29
11t273	CREAVEY-TEFLON	K2	2B	0
11t274	CREAVEY-TEFLON	K3	3B	0
11t275	CREAVEY-TEFLON	1B	1B	19
11t276	CREAVEY-TEFLON	2B	2B	10
11t277	CREAVEY-TEFLON	3B	3B	-40
11t281	CREAVEY-TEFLON	1B	1B	0
11t282	CREAVEY-TEFLON	2B	2B	-80
11t283	CREAVEY-TEFLON	3B	3B	-90
11t329	CREAVEY-TEFLON	1B	1B	-51
11t330	CREAVEY-TEFLON	2B	2B	-60
11t331	CREAVEY-TEFLON	3B	3B	-71

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t332	CREAVEY-TEFLON	1B	1B	-41
11t333	CREAVEY-TEFLON	2B	2B	-89
11t334	CREAVEY-TEFLON	3B	3B	-48
11t335	CREAVEY-TEFLON	1B	1B	-20
11t336	CREAVEY-TEFLON	2B	2B	-90
11t337	CREAVEY-TEFLON	3B	3B	-90
11t199	E540-80	1A	1A	-11
11t200	E540-80	2A	2A	-31
11t201	E540-80	3A	3A	-30
11t328	E540-80	3B	3B	-60
11t205	E540-80	1A	1A	-49
11t206	E540-80	2A	2A	-39
11t207	E540-80	3A	3A	-49
11t326	E540-80	1B	1B	-30
11t327	E540-80	2B	2B	-61
11t190	E740-75	1A	1A	-61
11t191	E740-75	2A	2A	-81
11t192	E740-75	3A	3A	-49
11t196	E740-75	1A	1A	-60
11t197	E740-75	2A	2A	-50
11t198	E740-75	3A	3A	-50
11t240	EYPEL-1801-70	1A	1A	-80
11t241	EYPEL-1801-70	2A	2A	-80
11t242	EYPEL-1801-70	3A	3A	-60
11t226	F0953-70	1A	1A	-61
11t227	F0953-70	2A	2A	-85
11t228	F0953-70	3A	3A	-80
11t232	F0953-70	1A	1A	-71
11t233	F0953-70	2A	2A	-82
11t234	F0953-70	3A	3A	-61
11t287	F0953-70	1B	1B	-1
11t288	F0953-70	2B	2B	-51
11t289	F0953-70	3B	3B	-53
11t293	F0953-70	1B	1B	-20
11t294	F0953-70	2B	2B	-80
11t295	F0953-70	3B	3B	-80
11t323	F0953-70	1B	1B	-51
11t324	F0953-70	2B	2B	-80

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t325	F0953-70	3B	3B	-50
11t256	KALREZ	1A	1A	10
11t284	KALREZ	1B	1B	20
11t285	KALREZ	2B	2B	10
11t286	KALREZ	3B	3B	20
11t314	L0677-70	1B	1B	-10
11t315	L0677-70	2B	2B	-52
11t316	L0677-70	3B	3B	-71
11t181	L0677-75	1A	1A	-80
11t182	L0677-75	2A	2A	-85
11t183	L0677-75	3A	3A	-61
11t184	L0677-75	1A	1A	-45
11t185	L0677-75	2A	2A	-50
11t186	L0677-75	3A	3A	-90
11t243	NPTFE	1A	1A	-22
11t244	NPTFE	2A	2A	-78
11t245	NPTFE	3A	3A	-77
11t269	NPTFE	K1	1B	-17
11t270	NPTFE	K2	2B	-20
11t271	NPTFE	K3	3B	-90
11t359	R-0404-50	1A	1A	-68
11t360	R-0404-50	2B	2B	-60
11t361	R-0404-50	3B	3B	-60
11t362	R-0404-50	1A	1A	-70
11t363	R-0404-50	2B	2B	-70
11t364	R-0404-50	3B	3B	-50
11t353	R-0404-70	1A	1A	-68
11t354	R-0404-70	2B	2B	-50
11t355	R-0404-70	3B	3B	-60
11t356	R-0404-70	1A	1A	-50
11t357	R-0404-70	2B	2B	-60
11t358	R-0404-70	3B	3B	-40
11t371	R1429-70	1A	1A	-18
11t372	R1429-70	2B	2B	10
11t373	R1429-70	3B	3B	-20
11t374	R1429-70	1A	1A	-20
11t375	R1429-70	2B	2B	-30
11t376	R1429-70	3B	3B	-20

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t413	ROW-TEFLON-SILICONE	1A	1A	-48
11t414	ROW-TEFLON-SILICONE	2B	2B	-57
11t415	ROW-TEFLON-SILICONE	3B	3B	-40
11t416	ROW-TEFLON-SILICONE	1A	1A	-50
11t417	ROW-TEFLON-SILICONE	2B	2B	-60
11t418	ROW-TEFLON-SILICONE	3B	3B	-40
11t422	ROW-TEFLON-VITON	1A	1A	-50
11t423	ROW-TEFLON-VITON	2B	2B	-40
11t424	ROW-TEFLON-VITON	3B	3B	0
11t425	ROW-TEFLON-VITON	1A	1A	-50
11t426	ROW-TEFLON-VITON	2B	2B	-41
11t427	ROW-TEFLON-VITON	3B	3B	-50
11t296	S0383-70	1B	1B	-1
11t297	S0383-70	2B	2B	-41
11t298	S0383-70	3B	3B	-90
11t302	S0383-70	1B	1B	-10
11t303	S0383-70	2B	2B	-30
11t304	S0383-70	3B	3B	-52
11t320	S0383-70	1B	1B	-60
11t321	S0383-70	2B	2B	-40
11t322	S0383-70	3B	3B	-78
11t305	S604-70	1B	1B	-7
11t306	S604-70	2B	2B	-51
11t307	S604-70	3B	3B	-8
11t308	S604-70	1B	1B	-1
11t309	S604-70	2B	2B	-51
11t310	S604-70	3B	3B	-60
11t317	S604-70	1B	1B	-11
11t318	S604-70	2B	2B	-65
11t319	S604-70	3B	3B	-61
11t121	S613-60	1A	1A	-70
11t122	S613-60	2A	2A	-70
11t123	S613-60	3A	3A	-71
11t124	S613-60	1A	1A	-70
11t125	S613-60	2A	2A	-71
11t126	S613-60	3A	3A	-70
11t127	S613-60	1A	1A	-71
11t128	S613-60	2A	2A	-71

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t129	S613-60	3A	3A	-70
11t130	S613-60	1A	1A	-70
11t131	S613-60	2A	2A	-71
11t132	S613-60	3A	3A	-70
11t151	S613-60	1A	1A	-70
11t152	S613-60	2A	2A	-71
11t153	S613-60	3A	3A	-70
11t100	S899-50	1A	1A	-91
11t101	S899-50	2A	2A	-90
11t102	S899-50	3A	3A	-41
11t103	S899-50	1A	1A	-81
11t104	S899-50	2A	2A	-90
11t105	S899-50	3A	3A	-90
11t106	S899-50	1A	1A	-86
11t107	S899-50	2A	2A	-90
11t108	S899-50	3A	3A	-81
11t109	S899-50	1A	1A	-90
11t110	S899-50	2A	2A	-92
11t217	V747-75	1A	1A	10
11t218	V747-75	2A	2A	-20
11t219	V747-75	3A	3A	0
11t223	V747-75	1A	1A	10
11t224	V747-75	2A	2A	-10
11t225	V747-75	3A	3A	0
11t154	V835-75	1A	1A	-21
11t155	V835-75	2A	2A	-37
11t156	V835-75	3A	3A	-29
11t157	V835-75	1A	1A	-20
11t158	V835-75	2A	2A	-42
11t159	V835-75	3A	3A	-28
11t160	V835-75	1A	1A	20
11t161	V835-75	2A	2A	-39
11t162	V835-75	3A	3A	-27
11t163	V835-75	1A	1A	10
11t164	V835-75	2A	2A	-41
11t165	V835-75	3A	3A	-31
11t187	V835-75	1A	1A	-40
11t188	V835-75	2A	2A	-40

O-RING ID	O-RING COUMPOUND	TOP FLANGE ID	BOTTOM FLANGE ID	FAILURE TEMPERATURE (DEGREES F)
11t189	V835-75	3A	3A	-40
11t235	V835-75	T6	B1	-41
11t236	V835-75	T6	B1	-40
11t392	V835-75	1A	1A	-30
11t393	V835-75	2B	2B	-31
11t394	V835-75	3B	3B	-30
11t440	V835-75	1A	1A	-19
11t443	V835-75	1A	1A	17
11t446	V835-75	1A	1A	-19

APPENDIX E

HIGH-TEMPERATURE SEAL PERFORMANCE TEST PROCEDURE

This appendix contains the reviewed and approved formal procedure for conducting the high-temperature seal performance tests.

SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE and PQAPP

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Revision	A	A	A	A	A	[illegible]	A	A	A	A	A	A	A	A

Page	15	16	17	18	19	20	21	22
Revision	A	A	A	A	A	A	A	A

Prepared by:

W.B. Leisher, Tech. Director, 6643 — Date: 12/17/93

D.R. Bronowski, Test Coordinator, 6643 — Date: 12-17-93

Approved by:

M.C. Brady, Dept. Manager, 6643 — Date: 1/31/94

P.E. McConnell, Task Leader, 6643 — Date: 1/10/94

L.E. Martin, QA Coordinator, 6600 — Date: 2/10/94

SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE

Issue Summary and Distribution

Issue	Date	Prepared by	Purpose of Issue
A	10/93	W.B. Leisher	Original Test Procedure

Copy	Revision	Name	Dept.
1	A	M.C. Brady	6643
2	A	P.E. McConnell	6643
3	A	D.R. Bronowski	6643
4	A	W.B. Leisher	6643
5	A	L.E. Martin	6600

SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE

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SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE

1.0 PURPOSE

The high temperature O-ring tests are being conducted in support of the OCRWM Program to better characterize the behavior of several O-ring compounds at high tempe...ures.

This document describes the procedure to be followed while performing these tests. Attachment A is the Level III Project Quality Assurance Program Plan.

2.0 SCOPE

This procedure is intended to apply to the measurement and high temperature testing of O-rings made of several compounds. It also includes a description of potential hazards and their control methods.

3.0 RESPONSIBILITIES

The Technical Director is responsible for providing hardware design, training, for overseeing the test activities, and for verifying that this procedure is followed.

The Test Coordinator is responsible for being familiar with this procedure and for either conducting or assisting with the testing in accordance with this procedure.

The Task Leader is responsible for budgeting and Program Management for the tests.

4.0 EQUIPMENT REQUIRED

4.1 DATA ACQUISITION SYSTEM: A Hewlett-Packard 3852A Data Acquisition System shall be used in conjunction with a 44701A Multimeter plug-in to measure pressure transducer output and convert thermocouple readings to temperature. The 44701A shall be within its calibration interval as specified by the Sandia Standards Laboratory (SSL, Dept. 1044). The 3852A also uses a relay card to control the pneumatically operated valves in the piping system. The 3852A is controlled and monitored through the IEEE General Purpose Interface Bus (GPIB) from the H-P 9000 computer system.

4.2 DISPLACEMENT GAGES: Two Heidenhain MT12B probes (with VRZ 404 counters) shall be used to measure O-ring thickness and the Inside Circumference Fixture gap. The direct reading counters shall be read either manually or by an IBM PC computer over the GPIB. The Heidenhains shall be within their calibration intervals as specified by the SSL-Extension (Dept. 2485-5). The operating procedure is given later in this document.

4.3 DC POWER SUPPLY: A DC power supply shall be used to provide 24 VDC excitation for the pneumatic valve control board in the piping system.

4.4 RESIDUAL GAS ANALYZER (RGA): A Dycor RGA and pumping station shall be used to measure tracer gas leakage past the test seal. The analyzer shall be calibrated at time of use by taking readings from tracer gas leaks that are within their calibration intervals as specified by the SNL Primary Standards Department (1743). Units of leakage are Scm^3/s (cm^3 at 0 C, 1 atm). See Reference 5.

4.5 SHORE DUROMETER: A Shore Durometer (hardness tester), Model 71400, Serial # 92012, shall be used to determine the hardness before and after exposure for the test seal. The tester shall be checked at time of use with the Shore-A Test Block Set, Serial # 92032, which shall be within its calibration interval as specified by Shore. The operating procedure is given by Reference 4.

4.6 CONTROL/RECORDING COMPUTER: An H-P 9000 computer system is used to prompt the operator, talk to the GPIB and RS-232 devices, and to record and process data. The software is designed specifically for data acquisition for seal testing. Output is sent to the line printer and to a disk file. The program maintains a HISTORY file of commands given to the computer and a NOTE PAD file into which the operator enters comments as applicable.

4.7 PRESSURE GAGE: An MKS model 122A capacitance manometer/readout shall be used to measure tracer gas cavity pressure. The 122A shall be within its calibration interval as specified by the Sandia Standards Laboratory (SSL, Dept. 1744). The 122A reading shall be taken by the computer program.

4.8 CIRCUMFERENCE MEASURING DEVICE (CMD): This device, SNL drawing number R43818, is used to determine the inside circumference from which the inside diamter of the seal can be calculated before and after test. The operating procedure is given later.

4.9 TORQUE WRENCH: A torque wrench adjustable for use at 75-in lb shall be within its calibration interval as specified by the Mechanical Measurements Team (Dept. 2483-1).

4.10 THERMOCOUPLES: Type K thermocouples shall be used for indication-only temperature readings. Any thermocouple that differs from the average of the others by more than three degrees F at ambient temperature shall be discarded.

4.11 REFERENCE PUBLICATIONS: The factory instruction manuals for the equipment listed above shall be considered as part of this procedure. Other applicable publications are as follow:

1. ES&H STANDARD OPERATING PROCEDURE SEAL TEST LABORATORY, ROOM E, BUILDING 883, SP472679.

2. MSDS Book, Room E, Building 883

3. SNL CSDP Quality Assurance Manual, Rev. E, 1/29/91

4. ASTM D 2240-86, STANDARD TEST METHOD FOR RUBBER PROPERTY--DUROMETER HARDNESS

5. AVS RECOMMENDED PRACTICE FOR THE CALIBRATION OF MASS SPECTROMETERS FOR PARTIAL PRESSURE ANALYSIS, 1993

5.0 COMPUTERS

5.1 H-P 9000: The H-P 9000 system programs shall be used while conducting these tests. Operating instructions and program descriptions may be found at the Operator's console.

Databases (setup information) and test data for each test are stored on hard disc and shall be transfered to floppy disc files or tape for backup. Hard copies of the HISTORY and NOTEPAD files, data bases, and plots shall be made for inclusion in the Test Log Note Book. Tape back-ups of the H-P system programs, databases, and test data shall be stored in the Technical Director's or Project Leader's office.

5.2 IBM PS/2: The IBM is used to run small utility programs in support of the lab operations. It has a MICRO488A interface box to convert RS-232 to GPIB for control and communications with GPIB equipment.

The only IBM program used in conjunction with the Hot Tests is ORMEAS.EXE. This program is used to read the Heidenhain thickness gage to measure and record the thicknesses at four points on each of three TS's. It also asks the user to enter four hardness and one gap measurements for each of the TS's. The data are tabulated, then sent to the printer and an ASCII disc file with identical formats. The hardcopy shall be included in the Test Log Book.

This program must be run before a hot test since some of the information compiled on the hardcopy must be entered in the H-P system data bases for the test. It is also run to make the Post-test physical measurements.

ORMEAS.EXE is located in C:\HOTTEST. The source file, ORMEAS.BAS, written in QuickBasic 4.5, is also in C:\HOTTEST. A back-up copy of the source is on the HOT TEST MASTER floppy disc in the Technical Director's office.

6.0 HAZARDS

6.1 WEIGHT: An assembled flange weighs approximately 55 pounds. Safety shoes shall be worn at all times while working with or moving flanges.

6.2 COMPRESSED GAS: The DOT approved tracer gas (helium, neon, or argon) cylinders shall be moved with a bottle cart. For use, they shall be either kept in the cart or chained to a substantial structure. A calibrated relief valve shall be installed on the regulator to protect the system from overpressure. No more than two 250-cu ft cylinders shall be in the lab at one time. The entrance door shall remain open whenever the room is occupied.

6.3 CHEMICALS: Small quanities of denatured alcohol on wipes and Q-Tips are used to clean seals and surfaces. There is no liquid alcohol waste. The wall mounted exhaust fan shall be turned on whenever alcohol is in use. Used wipes and Q-Tips shall be disposed of as HAZARDOUS materials in accordance with the ES&H SOP. Used mechanical vacuum pump oil shall be bottled and disposed of in accordance with the ES&H SOP.

6.4 LIQUID NITROGEN: LN2 is used in the cold trap of the RGA. A maximum of 7 liters is permitted in the lab. A face shield, chemical goggles, and insulating gloves shall be worn when filling either the transfer dewar or the cold trap.

6.5 HOT ITEMS: Flanges and portions of their associated plumbing and instrumentation will be tested at temperatures as high as 550 F. Flanges shall not be removed from the oven until they have cooled below 200 F, and insulating gloves shall be worn if the flanges are significantly above room temperature.

7.0 TEST IDENTIFICATION

The test identification (TI) shall consist of the letters 2HTXX, where 2HT stands for two-hour hot, and XX is the unique two digit sequence number from the test matrix. The test seal (TS) shall be identified by the TI plus an extension which is the flange set serial number, ie., -1A, -3C, etc.

The TI shall appear on all documents and records dealing with that test.

8.0 SEAL PHYSICAL MEASUREMENTS

The seal physical measurement procedures are given below: If questions arise, stop operations and discuss the problems with the Technical Director, Test Coordinator, or Task Leader to find the best solution.

Verify that all instruments and hardness calibration blocks are within their calibration interval. If the test is being conducted for actual data, do not continue if any instrument has an expired calibration.

Clean both the TS and the secondary seal (SS) with Kimwipes and denatured alcohol. Dispose of used wipes as HAZARDOUS material in accordance with the ES&H SOP.

Inspect both seals carefully to eliminate any seals that may have flaws that could adversely affect the test.

Identify both seals with tags giving the TI. Store in clean plastic bags until needed.

Mark and number the TS at four equally spaced points on the outside diameter. These points will be used for hardness and thickness measurements before and after exposure.

8.1 SHORE HARDNESS: The type A indentor shall be used with the Shore Durometer to make the hardness measurement. The procedure to be followed is given in ASTM D 2240-86, STANDARD TEST METHOD FOR RUBBER PROPERTY--DUROMETER HARDNESS, Ref. 4, except that the measurement is made at the four marked points on the TS instead of five points on a flat specimen.

The measurments shall be made before and after the temperature exposure and the data recorded in the proper locations on a copy of the attached work sheet.

The data shall be entered into the lab IBM computer when the program ORMEAS.EXE is run to measure thickness.

8.2 INSIDE DIAMETER: The Circumference Measuring Device (CMD) shall be used to determine the inside circumference.

To set up the CMD, close the device until the spacer bar is just snug between the jaws. Install the Heidenhain probe so its travel is at the approximate midpoint. Following the Heidenhain instructions, key the dimension marked on the spacer bar into the VRZ 404 counter as an offset. Set the counter reading to increase as the gap increases. Open the jaws slightly and remove the spacer bar. The device is now ready for use.

If the Heidenhain gets bumped or moved during use, repeat the offset setting as given above.

Loop the TS over the device. Slowly increase the gap while monitoring the TS thickness with a hand-held 1" micrometer. When the first indication of thickness reduction (0.001-0.003") due to stretch is noted, record the Heidenhain reading in the proper location on a copy of the attached work sheet. Reduce the gap, slide the TS around the CMD approximately 90 deg and repeat the measurement. Reduce the gap and remove the TS.

The average of the two gap readings shall be entered into the lab IBM computer when the program ORMEAS.EXE is run to measure thickness. The program converts the gap measurement to inside diameter with the following equation:

ID = 7.95 + GAP * 2 / 3.14159 inches

8.3 THICKNESS: The thickness (taken in the direction of compression) of the TS shall be measured at the four marked points using a Heidenhain displacement gage and fixture. The IBM program ORMEAS.EXE, located in the lab IBM computer HOTTST directory, prompts the user on setting up the equipment and making the measurements and records the data on hardcopy and in a disc file.

Figure 1, following, shows sample output from ORMEAS.

Reclean the TS and return it to the plastic bag until needed for fixture assembly.

9.0 TEST FLANGE ASSEMBLY

The following procedure shall be used to assemble the flange fixtures for test:

Inspect the O-ring mating surfaces on the plates carefully. Have the plates refinished if nicks or scratches are found. Small imperfections may be smoothed with emery paper.

Clean the O-ring mating surfaces of the top and bottom plates throroughly with denatured alcohol. Wear eye protection during this work. Dispose of used wipes and Q-tips as HAZARDOUS material in accordance with the ES&H SOP.

Install the previously inspected, measured, and cleaned TS and SS O-rings in their grooves (inner and outer respectively). DO NOT LUBRICATE THE O-RINGS. Be certain that the O-rings are not twisted or distorted to cause the flash to contact the top plate.

Make a last check for foreign material that might interfere with proper sealing.

Gently place the top plate on the bottom plate in an orientation that aligns the bolt holes and where the tubing fittings are approximately parallel. Do not slide the top plate on the bottom--lift the top before moving.

Lubricate the 24 closure bolts with Never-Sez. Snug, then torque, the bolts in a criss-cross pattern. The torque wrench setting is 75-in lb.

Protect the vacuum fittings to prevent entrance of foreign material.

Loop the TS and SS ID tags over the top plate vacuum fitting to identify the assembly until installation in the oven.

Figure 1 SAMPLE OUTPUT FROM ORMEAS.EXE

11-02-1993 12:15:39
PRE-TEST DATA.
DISK FILE IS NAMED B:2HT00.PRE

SAMPLE PROGRAM RUN.
DATA ARE NOT REAL.

INSTRUMENTATION INFORMATION
HEIDENHAIN MT12B/VRZ 404, S/N'S 4039624/4180120A, FILE 2109,
CALIBRATION EXPIRES 5/15/94.
HEIDENHA... MT12B/VRZ 404, S/N'S 4010355/4039642A, FILE 2069,
CALIBRATION EXPIRES 5/15/94.
SHORE DUROMETER MODEL 71400, S/N 92012, A-TYPE INDENTOR
CALIBRATION BLOCKS S/N 92032, EXPIRE 10/28/94
TYPE K THERMOCOUPLE, INDICATION ONLY.

	SET					
FLANGE	C3		2B		1A	
POINT	THICK	HARD	THICK	HARD	THICK	HARD
1	0.2738	70	0.2733	75	0.1771	65
2	0.2785	68	0.2684	73	0.1771	65
3	0.2729	64	0.2669	71	0.1771	65
4	0.2735	67	0.2650	73	0.1771	65
AVG	0.2747	67	0.2684	73	0.1771	65
SSD	0.0026	3	0.0035	2	0.0000	0
TEMP C	21.3		21.2		21.2	
GAP IN	6.500		6.450		6.400	
IN-DIA	12.09		12.06		12.02	
MFGR	PARKER		RAINIER		CHICAGO G	
COMPOUND	V0884-75		R1429-70		XXXX-90	
BATCH	123456		114921		01010	
CURE DATE	3Q91		4Q90		1Q99	
SERIAL NO	2HT00-C3		2HT00-2B		2HT00-1A	
SHORE-A DATE	3/12/92		3/12/92		3/12/92	
SHORE-A TEMP	23		23.2		23.4	

SET C3 COMMENT
NOT A REAL SEAL.
SET 2B COMMENT
DUMMY DATA.
SET 1A COMMENT
READ SAME FEELER GAGES 4 TIMES.

TEST TECHNICIAN ______________________
WBL

10.0 INITIAL RGA CALIBRATION

Verify that all calibrated leaks to be used are within their certified interval as shown on the Calibration Label. If the test is being conducted for actual data, do not continue if any leak has an expired calibration.

Connect the RGA to the system and start the pumps. Observe the manifold pressure on the RGA. It should indicate no more than 2 millitorr. If it is higher, it probably means the connection is leaking. Fill the cold trap with LN2.

After the RGA has warmed up (about 30 min), manually start a scan on neon 22, the isotope with M/Z of 22. When the reading is stable, open the neon (Ne) calibrated leak and allow the reading to restabilize. The "TABCAL" value which will be entered into the H-P 9000 database for Ne is found as follows:

TABCAL = Calibrated Leak Value/Restabilized RGA reading

Repeat the previous step scanning helium, M/Z of 4, and the helium (He) calibrated leak. Calculate the TABCAL value to be entered in the H-P 9000 database for He.

Record the TABCAL values on the work sheet.

11.0 TEST OPERATIONS

The high temperature test procedures are given below: If questions arise, stop operations and discuss the problems with the Technical Director, Test Coordinator, or Task Leader to find the best solution.

Verify that all instruments are within their certified interval by checking the expiration date on the Calibration Label. If the test is being conducted for actual data, do not continue if any instrument has an expired calibration.

Following the instructions at the H-P 9000 control console, start the SEALS program and create (or copy from older tests) the calibration, port, RGA, and thermal databases for this test. The TABCAL values are entered into the RGA database at this time. Set the RGA DWELL to its minimum value.

Connect a He supply cylinder to the tracer gas inlet line. Set the pressure to about 5 psig. Purge the line.

For each fixture in turn:

Install the assembled flange into the oven. Keep the flange numbers in order from top to bottom. The oven slots are numbered as Ports 1 through 3, top to bottom. Remove the seal ID tag from the fixture fitting and hang it on the proper Port # test cavity hose OUTSIDE the oven when the hose is connected.

Connect the proper Port # tracer gas supply hose to the bottom plate of the fixture. Insert the proper Port # thermocouple into the hole on the side of the bottom plate.

Open the tracer cavity and test cavity to the rough pump, one at a time. Moni-- pressure to verify correct connection and to determine if large leaks are present. If connections are incorrect or leaks are noted, make necessary reconnections or repairs. Repeat the checks until both cavities pump down satisfactorily.

Install and check the next fixture.

Rough pump on all cavities for about 1 hour to clean-up and reduce background.

For each fixture in turn:

Start the thermal and leak acquire portions of the program for the fixture/Port that is to be assembly leak tested. Set the data scan interval to 1 second.

Valve the RGA into the test cavity, and backfill the tracer cavity with He to about 760 Torr. After about 15 seconds, evacuate the tracer cavity. Monitor the He trace on the H-P display for about 30 seconds for indication of a leak, a sharp rise. Enter a comment on the NOTE PAD that Fixture XX is receiving the assembly leak test. Write the reading in the NOTE PAD. Valve the test cavity to the rough pump for clean-up. If the fixture has a leak rate greater than 1 E -08 Scm^3/s, remove, disassemble, reclean, and reassemble with new seals.

Repeat for the remaining fixtures.

After all fixtures have been tested and found to be leaking less than 1 E -08 Scm^3/s, valve all cavities to the rough pumps and allow to clean-up for at least one hour.

Replace the He supply bottle with the Ne/He mixture. Set the regulator to about 5 psig, and purge the lines.

Start the oven to ramp up to test temperature. Set the H-P scan time to 5 minutes. Monitor the middle fixture temperature on the H-P display.

When the temperature is about half way to test temperature, begin the RGA calibrations for each Port/fixture.

For each fixture in turn:

Switch to the desired Port on the H-P. Set the H-P to monitor masses 4 and 22.

Enter a comment on the NOTE PAD that RGA calibration is next.

Open both the He and Ne calibrated leaks at the same time. The readings will go to a peak and then fall to a stable value. Enter this value on the NOTE PAD.

Repeat for the remaining fixtures.

When the test temperature is reached, each fixture will be given a quick leak check with Ne/He. Set the H-P scan time to 1 second and only monitor Ne and He (masses 22 and 4).

For each fixture in turn:

Select the Port for test.

Enter a comment on the NOTE PAD that initial high temperature leak check for this Port is beginning. Valve the RGA into the test cavity. When the reading is stable, backfill the tracer cavity with Ne/He to about 760 Torr. Evacuate the tracer cavity after about 15 seconds. Monitor the RGA readings on the H-P display for about 15 seconds for indication of a leak, a sharp rise in both traces within 10 seconds of injection. Write the readings in the NOTE PAD. Valve the test cavity to the rough pump.

Repeat for the remaining fixtures.

Continue pumping the tracer cavities during the 2-hour soak period. Set data scan interval to 2 minutes.

Monitor Ne/He from all fixture test cavities on the H-P display. The readings should continue to decrease as the permeated tracer is pumped out during the soak period.

At the end of the 2-hour soak period, test each fixture, top to bottom, as follows:

Select the Port for test. Display the Ne and He traces on the H-P. Set data scan interval to 1 second.

Enter a comment on the NOTE PAD that the high temperature leak check for this Port is beginning.

Valve the RGA into the test cavity. When the reading is stable, backfill the tracer cavity with Ne/He to about 760 Torr. A leak will be shown by a sharp rise in both traces. Write the Ne/He readings in the NOTE PAD. Permeation is shown by a slower rise and a time difference between the two traces. Continue the test until well into the permeation region. Note the permeation breakthru times for both gasses in the NOTE PAD. Evacuate the tracer cavity, and valve the test cavity to the rough pump.

Repeat for the remaining fixtures.

Set data scan interval to 60 minutes. When all fixtures have been tested, turn the oven off and allow to cool to room temperature--probably overnight. Continue rough pumping on all cavities during cooling.

For each fixture in turn:

Enter a comment on the NOTE PAD that the final ambient temperature leak check for this Port is beginning. Set the data scan interval to 10 seconds.

Recalibrate the RGA for Ne and He as given above.

Set the data scan interval to 1 second. Valve the RGA into the test cavity. When the reading is stable, backfill the tracer cavity with Ne/He to about 760 Torr. Monitor the He/Ne traces on the H-P for about 15 seconds for indication of a leak, sharp rises in both traces. Note the reading in the NOTE PAD. Valve both cavities to the rough pumps.

Repeat for the remaining fixtures.

Halt data acquisition for both thermal and leak.

Disconnect and remove the fixtures from the oven. Reattach the ID tags to the fixture. Disassemble and inspect the seals for evidence of damage. Make the physical measurements as given earlier and record the data. Attach the ID tags to the seals and place in plastic bags.

12.0 RECORDS

12.1 The Test Coordinator shall sign the printer output at the spaces provided.

12.2 Copies of all printer output, disk files, and the work sheet shall be given to the Task Leader.

12.3 Original printer output, disk files, and the work sheet shall be kept in the HOT TEST notebook in the Seal Lab for backup purposes. HISTORY and NOTE PAD file hardcopies shall be made and placed into the HOT TEST notebood.

13.0 DEVIATIONS

13.1 Deviations from this procedure shall be noted on a copy of the next page of this document and approved by the Technical Director and the Task Leader.

13.2 The approved form shall be filed with the Test printouts in the HOT TEST notebook.

SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE

Deviation Report

Date ______________________

By ______________________

Test ID ____________________________

Seal Serial Number __________________

Change ____

____

____

____

Reason for Change ____

____

____

____

Comments ____

____

____

Approved by:

Technical Director	Date	Task Leader	Date

14.0 WORK SHEET

The following two pages are a sample Work Sheet. Make copies as needed.

HIGH TEMPERATURE TEST WORK SHEET

TEST # ________________ DATE ________________

		FLANGE SET				INSTRUMENTS
MANUFACTURER						
COMPOUND						
BATCH						
CURE DATE						
INSIDE DIAMETER						Heidenhain MT12B/
PRE-TEST	GAP					VRZ 404, File 2069
____	INSIDE CIRCUM					Expires 5/15/94
TEMP	INSIDE DIAM					
POST-TEST	GAP					
____	INSIDE CIRCUM					
TEMP	INSIDE DIAM					
CROSS SECTION						Heidenhain MT12B/
PRE-TEST	0					VRZ 404, File 2109
	90					Expires 5/15/94
____	180					
TEMP	270					
POST-TEST	0					
	90					
____	180					
TEMP	270					

TABCAL VALUES He ________, Ne ________

SHORE HARDNESS						Shore Calibration Blocks S/N 92032 Expires 10/28/94
PRE-TEST	0					
	90					
____	180					
TEMP	270					
POST-TEST	0					
	90					
____	180					
TEMP	270					

LEAK RATE					Varian He Cal Leak 793351, File 2282 Expires 12/13/95
PRE-TEST (HE)					
____ TEMP ____ PRES					
TEST (NE)					VTI Neon Cal Leak 01263, File 11621 Expires 3/1/94
____ TEMP ____ PRES					
POST-TEST (HE)					
____ TEMP ____ PRES					

PERMEATION TIME				
HELIUM				
NEON				
____ TEMP ____ PRES				

REMARKS:

TEST TECHNICIAN ______________________________

ATTACHMENT A

Rev A

Sandia National Laboratories Cask Systems Development Program

Project Quality Assurance Program Plan

1. Project: High Temperature O-Ring Tests

2. Quality Level: 3

3. Is this Project QA Program Plan a subplan ? ☐ yes ☒ no

4. Quality Level of this subplan: ___

5. Scope of this Project QA Program Plan: (Describe) QA Plan covering determination of sealing performance of certain O-ring compounds during and after exposure to high temperature.

6. The QA requirements specified in the following procedures will apply:

	All	With Exception	Not Applicable
PD 1.1, Preparation and Control of Program Directives, Rev C,	☐	☐	☒
PD 1.2, Management Appraisals and Effectiveness Assessments, Rev C	☐	☐	☒
PD 1.3, Commitment Tracking, Rev C	☒	☐	☐
PD 1.4, Organization, Rev C	☐	☐	☒
PD 1.5, Incoming Program Correspondence, Rev B	☒	☐	☐
PD 2.1, Software Quality Assurance, Rev B	☐	☐	☒
PD 2.2, Performance Evaluation Test Facility Approval, Rev B	☐	☐	☒
PD 2.3, Task Definition Statements, Rev B	☒	☐	☐
PD 2.4, Software Quality Assurance for Existing Software, Rev B	☐	☐	☒
PD 2.5, Control of Processes, Rev C	☒	☐	☐
PD 2.7, Test Control, Rev E	☐	☒*	☐
PD 2.8, Control of Measuring and Test Equipment, Rev D	☒	☐	☐
PD 2.9, Handling, Storage, and Shipping, Rev B	☒	☐	☐
PD 2.10, Inspection, Rev C	☐	☒*	☐
PD 3.2, Preparation and Control of Procurement Documents, Rev D	☐	☒*	☐
PD 3.3, Document Control, Rev C	☒	☐	☐
PD 3.4, Records Management, Rev C	☒	☐	☐
PD 4.1, Indoctrination and Training, Rev D	☒	☐	☐
PD 5.1, Quality Information Reporting, Rev C	☐	☐	☒
PD 5.2, Significant Problem Reporting and Corrective Action, Rev C	☒	☐	☐
PD 5.3, Quality Audit, Rev C	☐	☐	☒
PD 5.4, Stop Work Request, Rev B	☐	☐	☒
PD 5.5, Auditor / Lead Auditor Qualification and Certification, Rev C	☐	☐	☒
PD 5.6, Quality Program Levels of Effort, Rev C	☒	☐	☐
PD 5.7, Qualification of Inspection and Test Personnel, Rev B	☒	☐	☐
PD 5.8, Control of Nonconforming Items, Rev D	☐	☐	☒
PD 5.9, Surveillance, Rev B	☐	☐	☒
PD 5.10, Trend Analysis, Rev A	☐	☐	☒
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

7. Exceptions: State the procedure, the requirement, the exception, and the justification.

* The PD will be used as guidance for Level 3.

☐ Additional pages attached

Prepared by: ______________________
SNL CSDP Task Leader / Date

Approved by: ______________________ 2/10/94
6600 QA Coordinator / Date

Approved by: ______________________ 1/31/94
SNL CSDP Department Manager/Date

SANDIA NATIONAL LABORATORY

TRANSPORTATION SYSTEMS DEVELOPMENT DEPARTMENT

HIGH TEMPERATURE O-RING TESTS

TEST PROCEDURE

Deviation Report

Date 1/31/94

By W.B. Leisher

Test ID 2HTXX (all)

Seal Serial Number ALL

Change 1. Use the RGA and the Ne/He mixture to perform the assembly leak test. 2. Add a leak check point at -40 F to be taken after the post-hot-test ambient point. 3. Use He for the leak rate measurement instead of Ne.

Reason for Change 1. To reduce replumbing and introduction of possible leaks. 2. To verify that the seal will still work at low temperature. 3. Permeation times to reach the 1 E-9 leak level are long enough that a leak can be detected with He.

Comments 3. Ne is slow to clean up after permeation, leading to higher than desired background readings. Ne is still useful for verifying leakage vs permeation.

Approved by:

Technical Director 3/16/94 Date

Task Leader 3/16/94 Date

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